Electronic Supplementary Material (ESI) for ChemComm.

Supporting Information

Palladium(II)-Catalyzed Vinylic Geminal Double C-H Activation and Alkyne Annulation Reaction: Synthesis of Pentafulvenes

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Contents Page

1. General Information

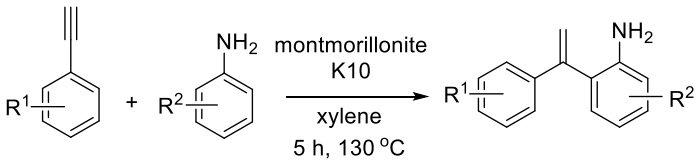
All the commercially available reagents were used as received. Melting points were measured with a Buchi B-540 melting point apparatus and are uncorrected. All experiments were monitored by thin layer chromatography. TLC was performed on Merck TLC Silica gel 60 F_{254} precoated plates. Column chromatography was performed on silica gel (100-200 mesh, Merck). NMR spectra were recorded on Bruker Avance III 500 MHz FTNMR spectrometer using tetramethylsilane (TMS) as an internal standard. HRMS data were recorded by electron spray ionization with a Q-TOF mass analyzer.

2.Reaction Procedures

2.1.General procedure for the synthesis of *N*-(2-(1-phenylvinyl)phenyl)acetamide:

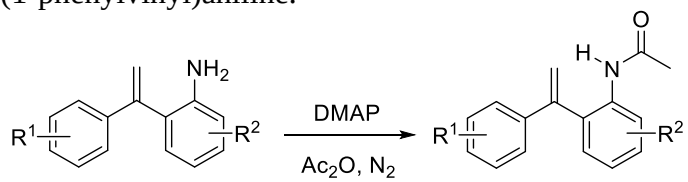
(i) Preparation of 2-(1-phenylvinyl)aniline:[1]

To a solution of phenylacetylene (10 mmol) and aniline (10 mmol) in xylene (10 mL), 1.0 g of montmorillonite K10 was added. Then, the reaction mixture was heated at 130 ^oC for 5 hours. After cooling to room temperature, the reaction mixture was filtered, the solvent was removed in vacuum and the residue was purified by column chromatography on silica gel to afford 2-(1-phenylvinyl)aniline derivative.



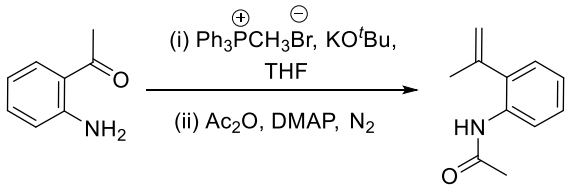
(ii) Preparation of *N*-(2-(1-phenylvinyl)phenyl)acetamide:[2]

A mixture of 2-(1-phenylvinyl)aniline (2.0 mmol) and 4-dimethylaminopyridine (30 mol %) in Ac_2O (6.0 mL) was stirred at room temperature under N_2 for 12 hours. Then Ac_2O was removed in vacuum and the residue was purified by column chromatography on silica gel to afford the acetyl derivative of 2-(1-phenylvinyl)aniline.

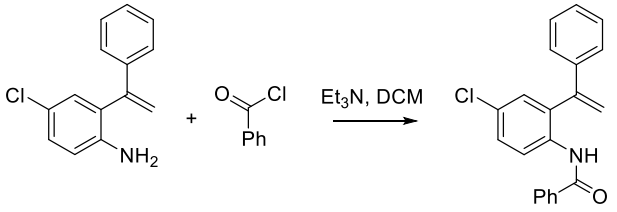


(iii) Preparation of *N*-(2-(prop-1-en-2-yl)phenyl)acetamide:[3] A mixture of KO^{t}Bu (622 mg, 5.55 mmol) and methyltriphenylphosphonium bromide (1.98 g, 5.55 mmol) was stirred at room temperature for 30 minutes in anhydrous THF (20 mL). Then, 2 aminoacetophenone (500 mg, 3.7 mmol, in 5 mL THF) was slowly added into the reaction mixture at 0 ^oC. After addition of the 2-aminoacetophenone, the reaction mixture was stirred at room temperature for four hours.

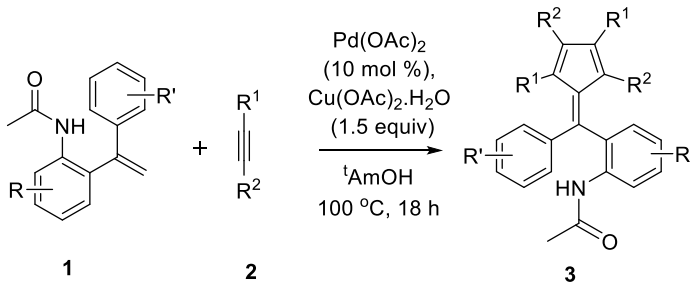
The solvent was removed under vacuo and water was added into the reaction mixture. Then it was extracted with ethyl acetate and the residue obtained by removal of the extract under vacuo was purified by column chromatography on silica gel to afford 2-(prop-1-en-2-yl)aniline (271 mg, 54%). This aniline (250 mg, 1.88 mmol) and 4-dimethylaminopyridine (30 mol %) in Ac_2O (5.0 mL) was stirred at room temperature under N_2 for 12 hours. Then, Ac_2O was removed in vacuum and the residue was purified by column chromatography on silica gel to afford *N*-(2-(prop-1-en-2-yl)phenyl)acetamide (280 mg, 85%).



(iv) Preparation of *N*-(4-chloro-2-(1-phenylvinyl)phenyl)benzamide:[4] Benzoyl chloride (168 mg, 1.2 mmol) was added dropwise to a stirring solution of 4-chloro-2-(1-phenylvinyl)aniline (229 mg, 1.0 mmol) in anhydrous DCM under nitrogen. Then, Et_3N (101 mg, 1.0 mmol) was added to the reaction mixture at room temperature and the resulting reaction mixture was allowed to stir for 18 hours. The reaction mixture was then extracted with dichloromethane (25 mL x 2) and the organic layer was washed with $NaHCO_3$ solution. The organic layer was dried over anhydrous Na_2SO_4, evaporated in vacuum and the residue was purified by column chromatography on silica gel to afford *N*-(4-chloro-2-(1-phenylvinyl)phenyl)benzamide (263 mg, 79%).



2.2. General procedure for the synthesis of pentafulvenes



A solution of phenylvinylacetamide (**1**, 0.5 mmol), alkyne (**2**, 1.0 mmol), $Cu(OAc)_2.H_2O$ (0.75 mmol) and $Pd(OAc)_2$ (10 mol %) in ^{t}AmOH (4 mL) was stirred at 100 ^{o}C under open air for 18 hours. The solvent was removed under vacuo and the crude reaction mixture was poured into water and extracted with ethyl acetate (25 mL x 3). The ethyl acetate layer was then washed with brine. Finally, it was dried over anhydrous Na_2SO_4 and the solvent was removed under vacuo. The crude product obtained was purified by silica gel (100-200 mesh) column chromatography using 25-30% EtOAc in Hexane as the eluent to afford pentafulvene **3**.

2.3. Procedure for the gram scale synthesis of pentafulvene 3ha:

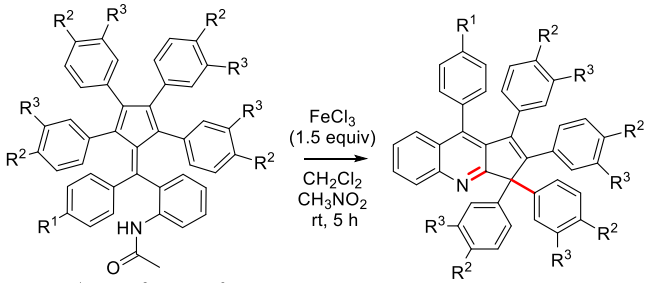
A solution of phenylvinylacetamide **1h** (1.0 g, 3.69 mmol), alkyne **2a** (1.3 g, 7.38 mmol), $Cu(OAc)_2.H_2O$ (1.1 g, 5.54 mmol) and $Pd(OAc)_2$ (83 mg, 0.37 mmol) in ^{t}AmOH (20 mL) was stirred at 100 ^{o}C under open air for 18 hours. The solvent was removed under vacuo and the crude reaction mixture was poured into water and extracted with ethyl acetate (50 mL x 4). The ethyl acetate layer was then washed with brine. Finally, it was dried over anhydrous Na_2SO_4 and the solvent was removed under vacuo. The crude product obtained was purified by silica gel (100-200 mesh) column chromatography using 25% EtOAc in Hexane as the eluent to afford pentafulvene **3ha** (1.52 g, 66%).

2.4. Procedure for the one pot synthesis of cyclopenta[*b*]quinoline 4ab:

A solution of phenylvinylacetamide **1a** (119 mg, 0.5 mmol), alkyne **2b** (206 mg,1.0 mmol), $Cu(OAc)_2.H_2O$ (150 mg, 0.75 mmol) and $Pd(OAc)_2$ (11 mg, 10 mol%) in ^{t}AmOH (4 mL) was stirred at 100 ^{o}C under open air for 18 hours. Then, the reaction was allowed to cool down to room temperature and $FeCl_3$ (122 mg, 0.75 mmol) was added into the reaction mixture. It was then stirred at room temperature for 5 hours. The solvent was removed under the vacuo, water was added and the mixture was extracted with CH_2Cl_2 (25 mL x 3). This solvent extract was dried over anhydrous Na_2SO_4 and it was then removed under vacuo. The crude product obtained was purified by silica gel (100-200 mesh) column chromatography using 3% EtOAc in Hexane as the eluent to afford cyclopenta[*b*]quinoline **4ab** (170 mg, 56%).

2.5. Procedure for the transformation of 3ab to 4ab by bromination reaction: To a stirred solution of compound **3ab** (38 mg, 0.06 mmol) in $CHCl_3$ (5 mL), Br_2 (11 mg, 0.07 mmol, diluted in $CHCl_3$) was added. The reaction mixture was allowed to stir for 8 hours at room temperature. The solvent was removed under vacuo and the crude product obtained was purified by silica gel (100-200 mesh) column chromatography using 3% EtOAc in Hexane as the eluent to afford cyclopenta[*b*]quinoline **4ab** (27 mg, 77%).

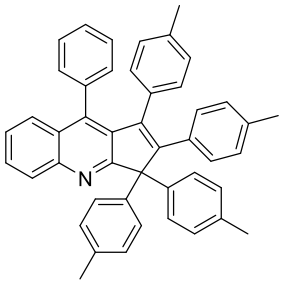
3.Transformation of pentafulvene derivatives(3ab-ad,3af,3ba-ca) to 3*H*-cyclopenta[*b*]quinolines (4ab-ad,4af,4ba-ca):



3ab (R^1 = H, R^2 = Me, R^3 = H)
3ac (R^1 = H, R^2 = OMe, R^3 = H)
3ad (R^1 = H, R^2 = F, R^3 = H)
3af (R^1 = H, R^2 = H, R^3 = Me)
3ba (R^1 = Me, R^2 = H, R^3 = H)
3ca (R^1 = Ph, R^2 = H, R^3 = H)

4ab (R^1 = H, R^2 = Me, R^3 = H, 90%)
4ac (R^1 = H, R^2 = OMe, R^3 = H, 79%)
4ad (R^1 = H, R^2 = F, R^3 = H, 89%)
4af (R^1 = H, R^2 = H, R^3 = Me, 88%)
4ba (R^1 = Me, R^2 = H, R^3 = H, 86%)
4ca (R^1 = Ph, R^2 = H, R^3 = H, 90%)

(a) Synthesis of 9-phenyl-1,2,3,3-tetra-*p*-tolyl-3*H*-cyclopenta[*b*]quinoline (4ab):[5] To a stirred solution of **3ab** (19 mg, 0.030 mmol) in anhydrous CH_2Cl_2 (2.0 mL) and CH_3NO_2 (0.5 mL), $FeCl_3$ (7 mg, 0.045 mmol) was added. The mixture was stirred at room temperature for 5 h. Water was added and the mixture was extracted with CH_2Cl_2. The organic phase was dried, concentrated and purified by silica gel column chromatography using 3% EtOAc in Hexane as the eluent to afford the title compound in 90% yield (white solid, 16 mg).

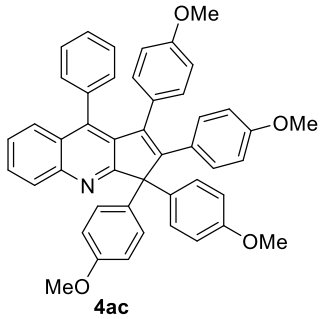


4ab

M.p.: 245-246 ^oC.^1H NMR (500 MHz, $CDCl_3$) δ 2.13 (s, 3H), 2.16 (s, 3H), 2.30 (s, 6H), 6.58 (d, *J* = 7.8 Hz, 2H), 6.64 (d, *J* = 8.0 Hz, 2H), 6.68-6.72 (m, 4H), 6.94-6.95 (m, 2H),7.00 (t, *J* = 7.7 Hz, 2H), 7.05 (d, *J* = 8.1 Hz, 4H), 7.09-7.12 (m, 1H), 7.29-7.32 (m, 1H), 7.38 (d, *J* = 8.2 Hz, 4H), 7.51-7.54 (m, 2H), 8.04 (d, *J* = 7.9 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 20.9, 21.0, 68.1, 125.6, 125.8, 126.4, 127.1, 127.5, 127.7, 127.9, 128.5, 129.3, 129.4, 129.5, 130.2, 130.5, 132.5, 132.6, 134.8, 135.2, 136.2, 138.5, 139.5, 140.9, 146.2, 151.8, 172.7. HRMS Calcd (ESI) m/z for $C_{46}H_{38}N$: $[M+H]^+$ 604.3004, found: 604.3008. Anal. Calcd for $C_{46}H_{37}N$: C, 91.50; H, 6.18; N, 2.32. Found: C, 91.77; H, 6.37; N, 2.06. IR ($CHCl_3$): 1584, 1511, 1220 cm^{-1}.

(b) Synthesis of 1,2,3,3-tetrakis(4-methoxyphenyl)-9-phenyl-3*H*-cyclopenta[*b*]quinoline (4ac):[5] To a stirred solution of **3ac** (21 mg, 0.030 mmol) in anhydrous CH_2Cl_2 (2.0 mL) and CH_3NO_2 (0.5 mL), $FeCl_3$ (7 mg, 0.045 mmol) was added. The mixture was stirred at room temperature for 5 h. Water was added and the mixture was extracted with CH_2Cl_2. The organic

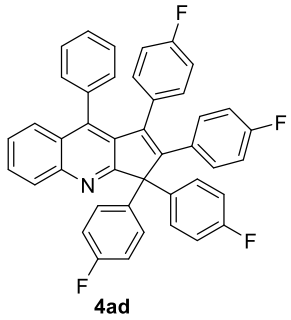
phase was dried, concentrated and purified by silica gel column chromatography using 3% EtOAc in Hexane as the eluent to afford the title compound in 79% yield (white solid, 16 mg).



4ac

M.p.: 212-214 ^{o}C.^{1}H NMR (500 MHz, $CDCl_3$) δ 3.64 (s, 3H), 3.69 (s, 3H), 3.77 (s, 6H), 6.35 (d, *J* = 8.7 Hz, 2H), 6.46 (d, *J* = 9.0 Hz, 2H), 6.67 (d, *J* = 8.7 Hz, 2H), 6.73 (d, *J* = 9.0 Hz, 2H), 6.79 (d, *J* = 8.9 Hz, 3H), 6.97 (d, *J* = 8.3 Hz, 2H), 7.06 (t, *J* = 7.7 Hz, 2H), 7.13 (t, *J* = 6.8 Hz, 1H), 7.31 (t, *J* = 7.6 Hz, 1H), 7.40 (d, *J* = 8.9 Hz, 5H), 7.53 (t, *J* = 7.6 Hz, 2H), 8.05 (d, *J* = 8.0 Hz, 1H).^{13}C NMR (125 MHz, $CDCl_3$) δ 54.8, 55.1, 67.2, 112.5, 112.9, 113.2, 125.6, 125.8, 126.8, 127.2, 127.5, 127.7, 128.0, 129.4, 130.3, 130.5, 130.6, 131.8, 132.3, 133.6, 134.8, 139.4, 139.8, 146.2, 151.8, 157.5, 158.0, 158.3, 172.9. HRMS Calcd (ESI) m/z for $C_{46}H_{38}NO_4$: $[M+H]^+$ 668.2801, found: 668.2802. Anal. Calcd for $C_{46}H_{37}NO_4$: C, 82.73; H, 5.58; N, 2.10. Found: C, 82.90; H, 5.64; N, 2.00. IR ($CHCl_3$): 1592, 1497, 1220 cm^{-1}.

(c) Synthesis of 1,2,3,3-tetrakis(4-fluorophenyl)-9-phenyl-3*H*-cyclopenta[*b*]quinoline (4ad): To a stirred solution of **3ad** (20 mg, 0.030 mmol) in anhydrous CH_2Cl_2 (2.0 mL), and CH_3NO_2 (0.5 mL), $FeCl_3$ (7 mg, 0.045 mmol) was added. The mixture was stirred at room temperature for 5 h. Water was added and the mixture was extracted with CH_2Cl_2. The organic phase was dried, concentrated and purified by silica gel column chromatography using 3% EtOAc in Hexane as the eluent to afford the title compound in 89% yield (white solid, 17 mg).

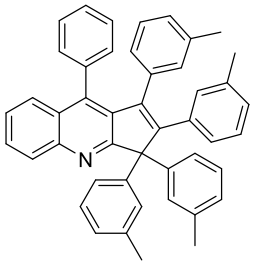


4ad

M.p.: 226-228 ^{o}C.^{1}H NMR (500 MHz, $CDCl_3$) δ 6.51 (t, *J* = 8.6 Hz, 2H), 6.65-6.71 (m, 6H), 6.94-7.00 (m, 6H), 7.09 (t, *J* = 7.6 Hz, 2H), 7.19 (t, *J* = 7.8 Hz, 1H), 7.36-7.39 (m, 5H), 7.57-7.62 (m, 2H), 8.05 (d, *J* = 8.3 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 68.1, 114.3 (d, *J* = 21.4 Hz), 114.4 (d, *J* = 21.0 Hz), 115.0 (d, *J* = 21.0 Hz), 126.1 (d, *J* = 20.7 Hz), 127.3, 127.4, 128.5,

129.4, 130.2, 130.7 (d, *J* = 7.9 Hz),130.9 (d, *J* = 8.1 Hz),131.0 (d, *J* = 3.4 Hz), 132.0 (d, *J* = 7.8 Hz), 134.3, 136.5 (d, *J* = 3.3 Hz), 140.5, 141.0, 146.4, 151.1, 161.1 (d, *J* = 244.8 Hz), 161.6 (d, *J* = 251.0 Hz), 161.8 (d, *J* = 244.9 Hz), 171.4. HRMS Calcd (ESI) m/z for $C_{42}H_{26}F_4N$: $[M+H]^+$ 620.2001, found: 620.2004. Anal. Calcd for $C_{42}H_{25}F_4N$: C, 81.41; H, 4.07; N, 2.26. Found: C, 81.56; H, 4.20; N, 1.90. IR ($CHCl_3$): 1587, 1495, 1219 cm^{-1}.

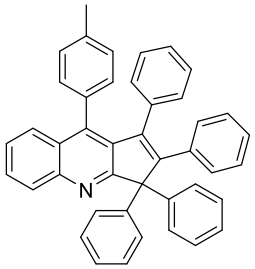
(d) Synthesis of 9-phenyl-1,2,3,3-tetra-*m*-tolyl-3*H*-cyclopenta[*b*]quinoline (4af): To a stirred solution of **3af** (19 mg, 0.030 mmol) in anhydrous CH_2Cl_2 (2.0 mL) and CH_3NO_2 (0.5 mL), $FeCl_3$ (7 mg, 0.045 mmol) was added. The mixture was stirred at room temperature for 5 h. Water was added and the mixture was extracted with CH_2Cl_2. The organic phase was dried, concentrated and purified by silica gel column chromatography using 3% EtOAc in Hexane as the eluent to afford the title compound in 88% yield (white solid, 16 mg).



4af

M.p.: 151-153 ^oC.^1H NMR (500 MHz, $CDCl_3$) δ 1.96-1.98 (s, 6H), 2.25-2.29 (s, 6H), 6.41 (s, 1H), 6.50 (s, 1H), 6.59 (d, *J* = 7.2 Hz, 1H), 6.63-6.67 (m, 2H), 6.72-6.81 (m, 4H), 6.88 (s, 2H), 7.02 (s, 2H), 7.06-7.09 (m, 2H), 7.17 (s, 4H), 7.32-7.35 (m, 3H), 7.54-7.57 (m, 2H), 8.08 (d, *J* = 8.8 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 21.0, 21.1, 21.5, 68.8, 125.6, 125.8, 126.5, 126.6, 126.8, 126.9, 127.0, 127.3, 127.5, 127.8, 129.5, 130.3, 130.4, 131.5, 132.3, 134.7, 135.2, 135.4, 135.9, 136.2, 139.8, 141.2, 146.2, 152.4, 172.3. HRMS Calcd (ESI) m/z for $C_{46}H_{38}N$: $[M+H]^+$ 604.3004, found: 604.3006. Anal. Calcd for $C_{46}H_{37}N$: C, 91.50; H, 6.18; N, 2.32. Found: C, 91.85; H, 6.27; N, 2.72. IR ($CHCl_3$): 1582, 1511, 1485, 1220 cm^{-1}.

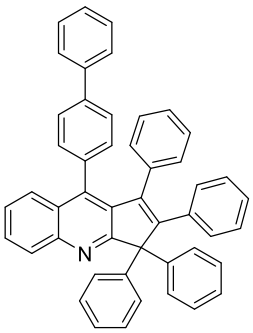
(e) Synthesis of 1,2,3,3-tetraphenyl-9-(*p*-tolyl)-3*H*-cyclopenta[*b*]quinoline (4ba): To a stirred solution of **3ba** (18 mg, 0.030 mmol) in anhydrous CH_2Cl_2 (2.0 mL) and CH_3NO_2 (0.5 mL), $FeCl_3$ (7 mg, 0.045 mmol) was added. The mixture was stirred at room temperature for 5 h. Water was added and the mixture was extracted with CH_2Cl_2. The organic phase was dried, concentrated and purified by silica gel column chromatography using 3% EtOAc in Hexane as the eluent to afford the title compound in 86% yield (white solid, 14 mg).



4ba

M.p.: 247-248 °C.^{1}H NMR (500 MHz, $CDCl_3$) δ 2.25 (s, 3H), 6.72-6.81 (m, 8H), 6.86-6.91 (m, 5H), 6.94-6.98 (m, 1H), 7.22-7.27 (m, 6H), 7.32-7.35 (m, 1H), 7.47-7.49 (m, 4H), 7.53-7.57 (m, 1H), 7.63 (d, *J* = 8.5 Hz, 1H), 8.05 (d, *J* = 8.4 Hz, 1H).^{13}C NMR (125 MHz, $CDCl_3$) δ 21.2, 69.4, 125.5, 125.8, 126.1, 126.9, 127.0, 127.1, 127.6, 127.9, 128.0, 128.1, 129.5, 129.6, 129.7, 130.2, 130.7, 131.6, 132.4, 135.5, 135.8, 136.7, 140.3, 141.4, 141.8, 146.5, 152.2, 172.3. HRMS Calcd (ESI) m/z for $C_{43}H_{32}N$: $[M+H]^+$ 562.2535, found: 562.2532. Anal. Calcd for $C_{43}H_{31}N$: C, 91.94; H, 5.56; N, 2.49. Found: C, 91.70; H, 5.61; N, 2.52. IR ($CHCl_3$): 1590, 1497, 1220 cm^{-1}.

(f) **Synthesis of 9-([1,1'-biphenyl]-4-yl)-1,2,3,3-tetraphenyl-3*H*-cyclopenta[*b*]quinoline[(4ca):** To a stirred solution of **3ca** (20 mg, 0.030 mmol) in anhydrous CH_2Cl_2 (2.0 mL) and CH_3NO_2 (0.5 mL), $FeCl_3$ (7 mg, 0.045 mmol) was added. The reaction mixture was stirred at room temperature for 5 h. Water was added and the mixture was extracted with CH_2Cl_2. The organic phase was dried, concentrated and purified by silica gel column chromatography using 3% EtOAc in Hexane as the eluent to afford the title compound in 90% yield (white solid, 17 mg).



4ca

M.p.: 245-248 °C.^{1}H NMR (500 MHz, $CDCl_3$) δ 6.74-6.80 (m, 5H), 6.88-6.92 (m, 2H), 6.96-6.99 (m, 1H), 7.07 (d, *J* = 8.2 Hz, 2H), 7.21-7.30 (m, 9H), 7.36-7.41 (m, 2H), 7.46-7.54 (m, 9H), 7.57-7.61 (m, 1H), 7.71 (d, *J* = 8.4 Hz, 1H), 8.08 (d, *J* = 8.4 Hz, 1H).^{13}C NMR (125 MHz,

$CDCl_3$) δ 69.1, 125.7, 125.9, 126.0, 126.8, 126.9, 127.0, 127.2, 127.3, 127.9, 128.1, 128.7, 129.4, 129.6, 130.6, 132.4, 133.6, 135.4, 135.5, 139.7, 139.9, 141.0, 141.2, 141.5, 146.4, 152.4, 172.2. HRMS Calcd (ESI) m/z for $C_{48}H_{34}N$: $[M+H]^+$ 624.2691, found: 624.2693. Anal. Calcd for $C_{48}H_{33}N$: C, 92.42; H, 5.33; N, 2.25. Found: C, 92.76; H, 5.30; N, 2.49. IR ($CHCl_3$): 1592, 1511, 1497,1221 cm^{-1}.

4. Measurement of fluorescence quantum yield (Φ_F):

Fluorescence quantum yields (Φ_F) of the compounds **4ab-ad**,**4af**,**4ba-ca** were calculated using quinine sulfate (in 0.5 M H_2SO_4 solution) as a standard (Φ_F = 0.54). Emission spectra of these compounds were recorded from 340 to 620 nm with excitation at 320 nm. Absorbance (optical density, OD) of all these samples were recorded at 320 nm and quantum yields were calculated according to equation (1), in which Φ_{ref} is the quantum yield of the reference, A_{sample} and A_{ref} are the areas under the emission spectra of the samples **4ab-ad**,**4af**,**4ba-ca** and the reference, OD_{ref} and OD_{sample} are the absorbances of the reference and the sample which were measured at the excitation wavelength; n_{sample} and n_{ref} are the refractive indices of the sample and the reference in solution.

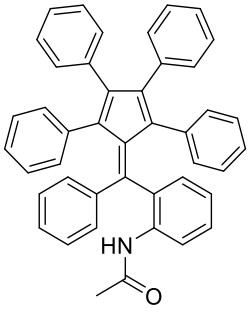
Sl. No	Compound	λ_{max} (nm)	λ_{em} (nm)	Φ_F
1	**4ab**	323	405	0.59
2	**4ac**	327	431	0.12
3	**4ad**	318	399	0.10
4	**4af**	320	431	0.14
5	**4ba**	320	406	0.11
6	**4ca**	321	410	0.16

$$\boldsymbol{\Phi F} = \boldsymbol{\Phi ref}\left(\frac{\boldsymbol{Asample}}{\boldsymbol{Aref}}\right)\boldsymbol{x}\left(\frac{\boldsymbol{ODref}}{\boldsymbol{ODsample}}\right)\boldsymbol{x}\left(\frac{\boldsymbol{nref}}{\boldsymbol{nsample}}\right)^2 \qquad (1)$$

References:

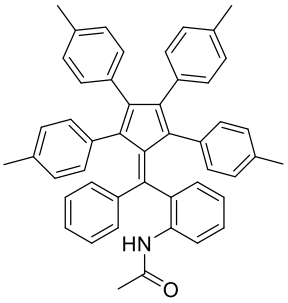
1. (a) X. Zhang, T.-L. Wang, C-D. Huo, X-C. Wang and Z.-J. Quan, *Chem. Commun.*, 2018, **54**, 3114; (b) T.-L. Wang, X.-J. Liu, C.-D. Huo, X.-C. Wang and Z.-J. Quan, *Chem. Commun.*, 2018, **54**, 499.
2. K. Kazuhiro, M. Kazuna, M. Osamu and K. Hisatoshi, *Bull. Chem. Soc. Jpn.*, 2005, **78**, 886.
3. (a) A. Boelke, L. D. Caspers and B. J. Nachtsheim, *Org. Lett.*, 2017, **19**, 5344. (b) L. D. Caspers, P. Finkbeiner and B. J. Nachtsheim, *Chem. Eur. J.*, 2017, **23**, 2748.
4. M. Chaitanya and P. Anbarasan, *Org. Lett.*, 2018, **20**, 1183.
5. D. Li and F. Zeng, *Org. Lett.*, 2017, **19**, 6498.

5. Spectral and Analytical Data:



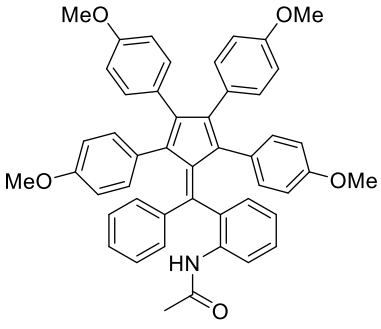
3aa

***N*-(2-(phenyl(2,3,4,5-tetraphenylcyclopenta-2,4-dien-1-ylidene)methyl)phenyl)acetamide (3aa):** A solution of *N*-(2-(1-phenylvinyl)phenyl)acetamide **1a** (237 mg, 1.0 mmol), diphenylacetylene (**2a**, 356 mg, 2.0 mmol), $Cu(OAc)_2.H_2O$ (300 mg, 1.5 mmol) and $Pd(OAc)_2$ (22 mg, 10 mol %) in ^{t}AmOH (8.0 mL) was stirred at 100 ^{o}C under open air for 18 hours. The solvent was removed under vacuo and the crude reaction mixture was poured into water and extracted with ethyl acetate (40 mL x 3). The ethyl acetate layer was then washed with brine. Finally, it was dried over anhydrous Na_2SO_4 and the solvent was removed under vacuo. The crude product obtained was purified by silica gel (100-200 mesh) column chromatography using 25% EtOAc in Hexane as the eluent to afford pentafulvene **3aa** (red solid, 74% yield, 438 mg). M.p.: 204-207 ^{o}C.^{1}H NMR (500 MHz, $CDCl_3$) δ 1.85 (s, 3H), 6.67-6.70 (m, 4H), 6.72-6.77 (m, 12H), 6.94-7.05 (m, 11H) 7.11 (d, *J* = 6.7 Hz, 1H), 7.16 (bs, 1H), 7.64 (d, *J* = 8.1 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) 24.2, 121.5, 123.2, 124.7, 125.2, 126.1, 126.9, 127.0. 127.2, 127.5, 128.2, 129.1, 129.8, 130.2, 130.4, 131.0, 132.0, 132.8, 132.9, 133.9, 134.0, 134.4, 135.1, 135.3, 136.0, 136.8, 140.3, 145.2, 145.3, 151.3, 167.8. HRMS Calcd (ESI) m/z for $C_{44}H_{34}NO$: $[M+H]^+$ 592.2640, found: 592.2639. Anal. Calcd for $C_{44}H_{33}NO$: C, 89.31; H, 5.62; N, 2.37. Found: C, 89.67; H, 5.68; N, 2.70. IR ($CHCl_3$): 3408, 1680, 1518, 1442 cm^{-1}.



3ab

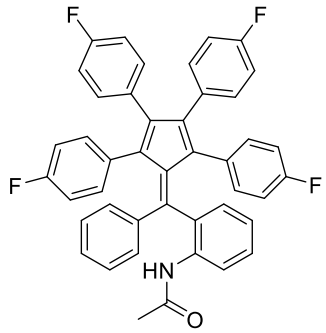
***N*-(2-(phenyl(2,3,4,5-tetra-*p*-tolylcyclopenta-2,4-dien-1-ylidene)methyl)phenyl)acetamide (3ab):** Following the general procedure **2.2**, the reaction was carried out by heating a mixture of *N*-(2-(1-phenylvinyl)phenyl)acetamide **1a** (119 mg, 0.5 mmol), 1,2-di-*p*-tolylethyne (**2b**, 206 mg, 1.0 mmol), $Pd(OAc)_2$ (11 mg, 10 mol %) and $Cu(OAc)_2.H_2O$ (150 mg, 0.75 mmol) in ^{t}AmOH (4.0 mL) at 100 ^{o}C for 18 h under air to afford the title compound in 76% yield (red solid, 246 mg). M.p.: 105-107 ^{o}C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.87 (s, 3H), 2.07 (s, 3H), 2.08 (s, 3H), 2.20 (s, 3H), 2.21 (s, 3H), 6.53 (d, *J* = 7.9 Hz, 2H), 6.56-6.65 (m, 10H), 6.70 (t, *J* = 7.5 Hz, 1H), 6.78-6.87 (m, 6H), 6.94-6.99 (m, 4H), 7.03 (d, *J* = 6.7 Hz, 1H), 7.24 (bs, 1H), 7.65 (d, *J* = 8.1 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 21.1, 21.5, 24.6, 121.6, 123.4, 127.4, 127.7, 127.9, 128.0, 128.7, 129.4, 130.5, 130.7, 131.3, 132.3, 132.6, 132.8, 133.0, 133.4, 133.5, 133.8, 134.0, 134.1, 134.2, 134.6, 135.7, 135.8, 136.2, 141.0, 145.2, 145.4, 146.2, 150.5, 167.9. HRMS Calcd (ESI) m/z for $C_{48}H_{42}NO$: $[M+H]^+$ 648.3266, found: 648.3268. Anal. Calcd for $C_{48}H_{41}NO$: C, 88.99; H, 6.38; N, 2.16. Found: C, 89.21; H, 6.49; N, 2.30. IR ($CHCl_3$): 3409, 1683, 1514, 1443 cm^{-1}.



3ac

***N*-(2-(phenyl(2,3,4,5-tetrakis(4-methoxyphenyl)cyclopenta-2,4-dien-1-ylidene)methyl)phenyl)acetamide (3ac):** Following the general procedure **2.2**, the reaction was carried out by heating a mixture of *N*-(2-(1-phenylvinyl)phenyl)acetamide (**1a**, 119 mg, 0.5 mmol), 1,2-bis(4-methoxyphenyl)ethyne (**2c**, 238 mg, 1.0 mmol), $Pd(OAc)_2$ (11 mg, 10 mol %) and $Cu(OAc)_2.H_2O$ (150 mg, 0.75 mmol) in ^{t}AmOH (4.0 mL) at 100 ^{o}C for 18 h under air to afford the title compound in 63% yield (red solid, 224 mg). M.p.: 73–77 ^{o}C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.85 (s, 3H), 3.61 (s, 3H), 3.62 (s, 3H), 3.68 (s, 3H), 3.70 (s, 3H),

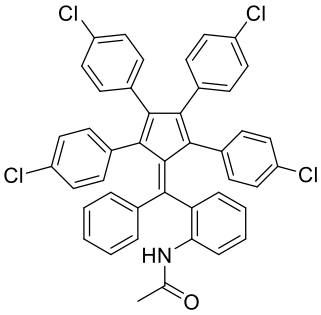
6.30-6.34 (m, 4H), 6.52-6.56 (m, 4H), 6.60-6.63 (m, 5H), 6.66 (d, *J* = 8.3 Hz, 2H), 6.72 (t, *J* = 7.3 Hz, 1H), 6.84-6.90 (m, 2H), 6.93-6.99 (m, 5H), 7.04 (d, *J* = 7.1 Hz, 1H), 7.18 (s, 1H), 7.65 (d, *J* = 8.2 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 24.2, 54.8, 54.9, 55.0, 112.4, 112.6, 112.7, 121.5, 123.2, 127.1, 127.4, 127.6, 127.8, 128.6, 128.7, 129.3, 129.5, 131.5, 131.7, 131.9, 132.1, 132.7, 133.1, 133.2, 133.6, 135.8, 140.6, 144.5, 144.6, 145.7, 149.5, 156.6, 156.9, 157.7, 167.6. HRMS Calcd (ESI) m/z for $C_{48}H_{42}NO_5$: $[M+H]^+$ 712.3063, found: 712.3060. Anal. Calcd for $C_{48}H_{41}NO_5$: C, 80.99; H, 5.81; N, 1.97. Found: C,80.86; H, 5.75; N, 2.08. IR ($CHCl_3$): 3401, 1687, 1502, 1441, 1244 cm^{-1}.



3ad

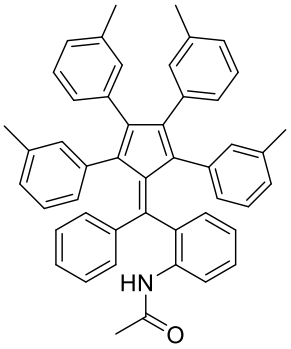
***N*-(2-(phenyl(2,3,4,5-tetrakis(4-fluorophenyl)cyclopenta-2,4-dien-1-ylidene)methyl)phenyl)acetamide (3ad):** Following the general procedure **2.2**, the reaction was carried out by heating a mixture of *N*-(2-(1-phenylvinyl)phenyl)acetamide **1a** (119 mg, 0.5 mmol), 1,2-bis(4-fluorophenyl)ethyne (**2d**, 214 mg, 1.0 mmol), $Pd(OAc)_2$ (11 mg, 10 mol %) and $Cu(OAc)_2.H_2O$ (150 mg, 0.75 mmol) in ^{t}AmOH (4.0 mL) at 100 ^{o}C for 18 h under air to afford the title compound in 78% yield (red solid, 259 mg). M.p.: 196–200 ^{o}C.^{1}H NMR (500 MHz, $CDCl_3$) δ 1.81 (s, 3H), 6.44-6.51 (m, 4H), 6.60-6.73 (m, 13H), 6.89 (t, *J* = 7.4 Hz, 1H), 6.95-7.00 (m, 3H), 7.05 (q, *J* = 8.1 Hz, 2H), 7.13 (d, *J* = 7.2 Hz, 1H), 7.32 (bs, 1H), 7.64 (d, *J* = 8.1 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 24.1, 113.9, 114.0 (d, *J* = 21.2 Hz), 114.1 (d, *J* = 20.5 Hz), 114.3 (d, *J* = 21.2 Hz), 122.1, 123.4, 126.4, 127.4, 127.9, 128.8, 129.5, 130.0, 130.6 (d, *J* = 3.2 Hz), 130.8 (d, *J* = 3.2 Hz), 131.7 (d, *J* = 7.7 Hz), 131.8 (d, *J* = 7.9 Hz), 132.4 (d, *J* = 3.2 Hz), 132.5 (d, *J* = 3.2 Hz), 133.0, 133.1, 133.6, 133.9, 136.0, 139.9, 144.1, 144.2, 144.4, 152.0, 160.3 (d, *J* = 243.6 Hz), 160.6

(d, J = 243.7 Hz), 161.2 (d, J = 245.6 Hz), 161.3 (d, J = 245.6 Hz), 167.4. HRMS Calcd (ESI) m/z for $C_{44}H_{30}F_4NO$: $[M+H]^+$ 664.2264, found: 664.2268. Anal. Calcd for $C_{44}H_{29}F_4NO$: C, 79.63; H, 4.40; N, 2.11. Found: C, 79.81; H, 4.27; N, 2.03. IR ($CHCl_3$): 3422, 1638, 1509, 1444, 1222 cm^{-1}.



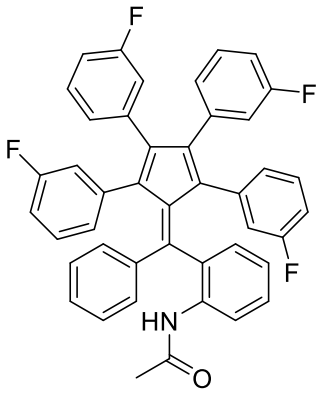
3ae

***N*-(2-(phenyl(2,3,4,5-tetrakis(4-chlorophenyl)cyclopenta-2,4-dien-1-ylidene)methyl)phenyl)acetamide (3ae):** Following the general procedure **2.2**, the reaction was carried out by heating a mixture of *N*-(2-(1-phenylvinyl)phenyl)acetamide (**1a**, 119 mg, 0.5 mmol), 1,2-bis(4-chlorophenyl)ethyne (**2e**, 247 mg, 1.0 mmol), $Pd(OAc)_2$ (11 mg, 10 mol %) and $Cu(OAc)_2.H_2O$ (150 mg, 0.75 mmol) in ^{t}AmOH (4.0 mL) at 100 ^{o}C for 18 h under air to afford the title compound in 68% yield (red solid, 247 mg). M.p.: 100-103 ^{o}C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.78 (s, 3H), 6.56-6.64 (m, 7H), 6.72-6.78 (m, 4H), 6.86-6.90 (m, 2H), 6.92-7.01 (m, 8H), 7.06–7.14 (m, 4H), 7.63 (d, J = 8.0 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 24.1, 122.3, 123.6, 127.3, 127.5, 127.6, 128.0, 128.3, 129.6, 129.8, 130.2, 130.9, 131.3, 131.4, 131.6, 132.0, 132.4, 132.5, 132.9, 133.0, 133.1, 133.4, 133.5, 134.0, 134.1, 134.2, 134.8, 136.1, 139.7, 143.6, 143.8, 143.9, 152.9, 167.6. HRMS Calcd (ESI) m/z for $C_{44}H_{30}Cl_4NO$: $[M+H]^+$ 728.1082, found: 728.1088. Anal. Calcd for $C_{44}H_{29}Cl_4NO$: C, 72.44; H, 4.01; N, 1.92. Found: C, 72.68; H, 4.30; N, 2.25.IR ($CHCl_3$): 3400, 1680, 1580, 1120 cm^{-1}.



3af

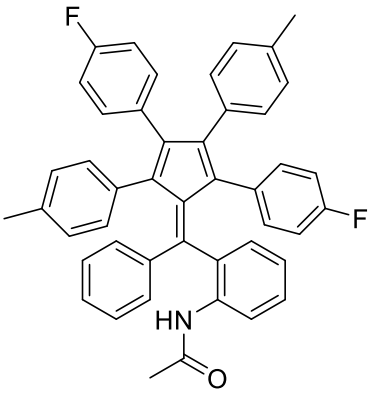
***N*-(2-(phenyl(2,3,4,5-tetra-*m*-tolylcyclopenta-2,4-dien-1-ylidene)methyl)phenyl)acetamide (3af):** Following the general procedure **2.2**, the reaction was carried out by heating a mixture of *N*-(2-(1-phenylvinyl)phenyl)acetamide (**1a**, 119 mg, 0.5 mmol), 1,2-di-*m*-tolylethyne (**2f**, 206 mg, 1.0 mmol), $Pd(OAc)_2$ (11 mg, 10 mol %) and $Cu(OAc)_2.H_2O$ (150 mg, 0.75 mmol) in ^{t}AmOH (4.0 mL) at 100 ^{o}C for 18 h under air to afford the title compound in 53% yield (red solid, 172 mg). M.p.: 101–102 ^{o}C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.85 (s, 3H), 1.93 (s, 6H), 1.94 (s, 6H), 6.41-6.50 (m, 11H), 6.54 (s, 1H), 6.57-6.60 (m, 2H), 6.64 (s, 1H), 6.74-6.81 (m, 5H), 6.86-6.88 (m, 5H), 7.66 (s, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 20.9, 21.0, 21.1, 21.2, 24.3, 122.9, 125.4, 125.9, 126.6, 126.7, 126.8, 127.0, 127.3, 127.4, 127.6, 128.2, 128.9, 129.5, 131.2, 131.3, 131.9, 132.4, 133.6, 133.7, 135.0, 135.2, 135.8, 135.9, 136.0, 136.6, 145.3, 150.4, 167.5. HRMS Calcd (ESI) m/z for $C_{48}H_{42}NO$: $[M+H]^+$ 648.3266, found: 648.3264. Anal. Calcd for $C_{48}H_{41}NO$:C, 88.99; H, 6.38; N, 2.16. Found C, 89.05; H, 6.33; N, 1.88. IR ($CHCl_3$): 3410, 1680, 1579, 1444 cm^{-1}.



3ag

***N*-(2-(phenyl(2,3,4,5-tetrakis(3-fluorophenyl)cyclopenta-2,4-dien-1-ylidene)methyl)phenyl)acetamide (3ag):** Following the general procedure **2.2**, the reaction was carried out by heating a mixture of *N*-(2-(1-phenylvinyl)phenyl)acetamide (**1a**, 119 mg, 0.5 mmol),1,2-bis(3-fluorophenyl)ethyne (**2g**, 214 mg, 1.0 mmol), $Pd(OAc)_2$ (11 mg, 10 mol %) and $Cu(OAc)_2.H_2O$ (150 mg, 0.75 mmol) in ^{t}AmOH (4.0 mL) at 100 ^{o}C for 18 h under air to afford the title compound in 77% yield (brown solid, 256 mg). M.p.: 173–176 ^{o}C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.78 (s, 3H), 6.39-6.44 (s, 3H), 6.46-6.56 (m, 6H), 6.72-6.79 (m, 5H), 6.90-7.08 (m, 10H), 7.22 (d, *J*

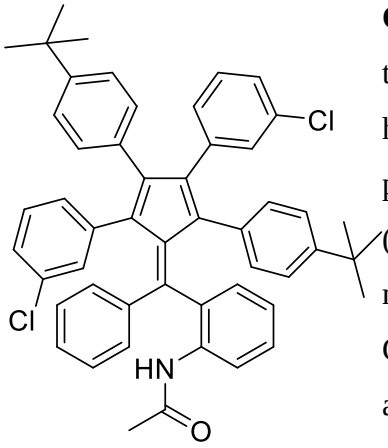
= 7.1 Hz, 1H), 7.68 (bs, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 24.0, 112.1 (d, *J* = 21.0 Hz), 112.5 (d, *J* = 20.9 Hz), 112.7, 113.6 (d, *J* = 21.4 Hz), 116.7 (d, *J* = 21.0 Hz), 116.9 (d, *J* = 21.0 Hz), 117.4 (d, *J* = 21.4 Hz), 121.9, 123.4, 125.8 (d, *J* = 21.0 Hz), 125.9 (d, *J* = 21.0 Hz), 126.0, 126.6, 127.5, 127.9, 128.6 (d, *J* = 8.4 Hz), 128.7 (d, *J* = 8.0 Hz), 128.8, 129.9, 130.5, 131.4, 132.2, 132.9, 133.6, 134.1, 136.1, 136.7 (d, *J* = 8.0 Hz), 136.8 (d, *J* = 8.0 Hz), 137.9, 138.0, 138.5 (d, *J* = 8.1 Hz), 139.7, 143.7, 144.0, 144.1, 155.7, 160.5 (d, *J* = 243.4 Hz), 161.6 (d, *J* = 243.5 Hz), 161.8 (d, *J* = 243.9 Hz), 167.5. HRMS Calcd (ESI) m/z for $C_{44}H_{30}F_4NO$: $[M+H]^+$ 664.2264, found: 664.2266. Anal. Calcd for $C_{44}H_{29}F_4NO$: C, 79.63; H, 4.40; N, 2.11. Found: C, 79.91; H, 4.70; N, 2.42. IR ($CHCl_3$): 3412, 1668, 1436 cm^{-1}.



3ah
(ratio of regioisomers: 7:7:1)

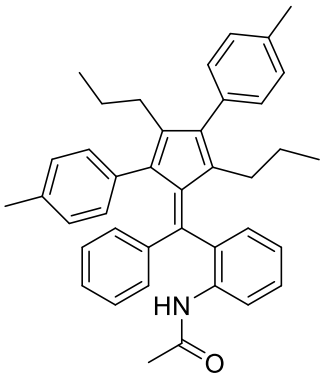
Compound 3ah (ratio of regioisomers: 7:7:1): Following the general procedure **2.2**, the reaction was carried out by heating a mixture of *N*-(2-(1-phenylvinyl)phenyl)acetamide (**1a**, 119 mg, 0.5 mmol), 1-fluoro-4-(*p*-tolylethynyl)benzene (**2h**, 210 mg, 1.0 mmol), $Pd(OAc)_2$ (11 mg, 10 mol %) and $Cu(OAc)_2.H_2O$ (150 mg, 0.75 mmol) in ^{t}AmOH (4.0 mL) at 100 °C for 18 h under air to afford the title compound in 60% yield (red solid, 197 mg). M.p.: 105–108 °C.^{1}H NMR (500 MHz, $CDCl_3$) δ 1.83 (s, 3H), 2.06 (s, 3H), 2.22 (s, 3H), 6.30-7.20 (m, 24H), 7.40-7.80 (s, 2H).^{13}C NMR (125 MHz, $CDCl_3$) δ 20.8, 21.1, 24.1, 24.2, 25.2, 29.6, 113.8, 113.9, 114.0, 114.1, 114.2, 121.4, 121.6, 121.8, 122.0, 123.2, 123.3, 123.4, 127.2, 127.3, 127.5, 127.6, 127.7, 127.8, 127.9, 128.2, 128.7, 129.1, 129.3, 129.4, 129.8, 130.1 (d, *J* = 19.7 Hz), 130.2 (d, *J* = 19.7 Hz), 130.3, 130.8, 131.2, 131.4, 131.8, 131.9, 132.0, 132.3, 132.4, 132.8, 133.4, 133.7, 133.8, 134.2, 134.5, 134.6, 134.7, 135.8 (d, *J* = 4.2 Hz), 135.9 (d, *J* = 4.4 Hz), 138.5, 140.2, 140.3, 140.4, 143.5, 143.8, 145.0, 145.2, 145.3, 145.4, 145.6, 145.7, 150.4, 151.0, 151.1,

151.7, 159.3, 159.5, 160.2 (d, J = 242.6 Hz), 160.5 (d, J = 242.9 Hz), 161.2, 161.2 (d, J = 244.4 Hz), 167.5, 169.1. HRMS Calcd (ESI) m/z for $C_{46}H_{36}F_2NO$: $[M+H]^+$ 656.2765, found: 656.2764. Anal. Calcd for $C_{46}H_{35}F_2NO$: C, 84.25; H, 5.38; N, 2.14. Found:C,84.09; H, 5.60; N, 1.88. IR ($CHCl_3$): 3410, 1676, 1579, 1512, 1220 cm^{-1}.



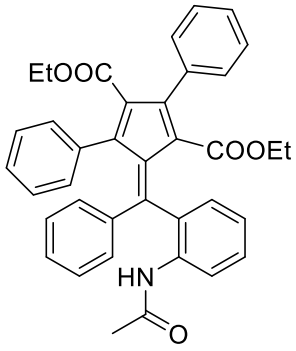
3ai
(ratio of regioisomers: 3:3:1):

Compound 3ai (ratio of regioisomers: 3:3:1): Following the general procedure **2.2**, the reaction was carried out by heating a mixture of *N*-(2-(1-phenylvinyl)phenyl)acetamide (**1a**, 119 mg, 0.5 mmol),1-((4-(*tert*-butyl)phenyl)ethynyl)-3-chlorobenzene (**2i**, 269 mg, 1.0 mmol), $Pd(OAc)_2$ (11 mg, 10 mol %) and $Cu(OAc)_2.H_2O$ (150 mg, 0.75 mmol) in ^{t}AmOH (4.0 mL) at 100 ^{o}C for 18 h under air to afford the title compound in 53% yield (red solid, 204 mg). M.p.: 102–106 ^{o}C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.14 (s, 9H), 1.22 (s, 9H), 1.82 (s, 3H), 6.50-6.72 (m, 9H), 6.72-6.84 (m, 3H), 6.85-7.65 (14H).^{13}C NMR (125 MHz, $CDCl_3$) δ 24.5, 31.3, 31.4, 34.4, 34.6, 34.7, 121.6, 122.0, 123.5, 124.2, 124.3, 124.4, 125.1, 125.6, 126.5, 126.6, 127.4, 127.6, 127.7, 127.8, 128.0, 128.4, 128.6, 128.8, 128.9, 129.1, 129.5, 129.6,129.8, 130.0, 130.1, 130.2, 130.3, 130.4, 130.6, 130.7, 130.9, 131.3, 131.6, 131.7, 131.8, 131.9, 132.1, 132.3, 132.6, 132.7, 132.8, 132.9, 133.0, 133.1, 133.2, 133.3, 134.0, 134.1, 135.0, 135.4, 135.5, 135.9, 136.2, 137.2, 137.4, 139.1, 140.5, 140.6, 142.9, 143.4, 145.3, 145.5, 146.1, 147.8, 148.1, 148.2, 149.6, 149.8, 152.2, 153.3, 167.8. HRMS Calcd (ESI) m/z for $C_{52}H_{48}Cl_2NO$: $[M+H]^+$ 772.3113, found: 772.3116. Anal. Calcd for $C_{52}H_{47}Cl_2NO$: C, 80.81; H, 6.13; N, 1.81. Found: C, 80.89; H, 6.03; N, 1.96. IR ($CHCl_3$): 3418, 1642, 1521 cm^{-1}.



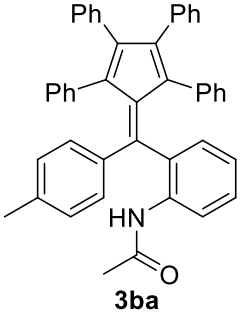
3aj
(ratio of regioisomers: 3:3:1:1)

Compound 3aj (ratio of regioisomers: 3:3:1:1): Following the general procedure **2.2**, the reaction was carried out by heating a mixture of *N*-(2-(1-phenylvinyl)phenyl)acetamide (**1a**, 119 mg, 0.5 mmol), 1-methyl-4-(pent-1-yn-1-yl)benzene (**2j**, 158 mg, 1.0 mmol), $Pd(OAc)_2$ (11 mg, 10 mol %) and $Cu(OAc)_2.H_2O$ (150 mg, 0.75 mmol) in ^{t}AmOH (4.0 mL) at 100 ^{o}C for 18 h under air to afford the title compound in 32% yield (orange solid, 88 mg). M.p.: 58-60 ^{o}C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.24-0.34 (m, 3H), 0.46-0.57 (m, 3H), 0.80-1.06 (m, 4H), 1.60-2.26 (m, 4H), 1.92-2.38 (s, 9H), 6.66-7.01 (m, 8H), 7.02-7.24 (m, 5H), 7.27-8.30 (m, 5H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 13.7, 13.9, 14.0, 20.8, 21.2, 22.4, 24.5, 24.7, 24.9, 25.3, 28.4, 28.5, 28.6, 28.9, 120.6, 120.7, 121.0, 122.9, 123.6, 123.7, 127.0, 127.1, 127.5, 127.6, 127.7, 127.8, 127.9, 128.2, 128.7, 128.8, 128.9, 129.4, 129.7, 131.2, 131.8, 132.0, 132.1, 132.2, 132.3, 132.4, 132.5, 132.6, 132.7, 132.9, 133.2, 133.8, 133.9, 134.1, 134.5, 134.6, 135.5, 135.9, 136.2, 136.3, 136.4, 140.8, 142.3, 145.2, 145.7, 146.0, 146.1, 146.2, 146.7, 147.1, 147.8, 167.6, 168.0. HRMS Calcd (ESI) m/z for $C_{40}H_{42}NO$: $[M+H]^+$ 552.3240, found: 552.3266. Anal. Calcd for $C_{40}H_{41}NO$: C, 87.07; H, 7.49; N, 2.54. Found: C, 87.19; H, 7.28; N, 2.77. IR ($CHCl_3$): 3400, 1638, 1582, 1220 cm^{-1}.



3ak
(ratio of regioisomers: 5:3:1)

Compound3ak, (ratio of regioisomers: 5:3:1): Following the general procedure **2.2**, the reaction was carried out by heating a mixture of *N*-(2-(1-phenylvinyl)phenyl)acetamide (**1a**, 119 mg, 0.5 mmol), ethyl 3-phenylpropiolate (**2k**, 174 mg, 1.0 mmol), $Pd(OAc)_2$ (11 mg, 10 mol %) and $Cu(OAc)_2.H_2O$ (150 mg, 0.75 mmol) in ^{t}AmOH (4.0 mL) at 100 ^{o}C for 18 h under air to afford the title compound in 54% yield (gum, 158 mg). ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.66-1.07 (m, 6H), 1.53 (s, 1.6H), 1.67 (s, 0.4H), 1.83 (s, 1H), 3.27-3.39 (m, 1.8H), 3.75-3.87 (m, 1.7H), 3.88-3.90 (m, 0.5H), 6.64-7.05 (m, 10H), 7.18 (t, *J* = 9.5 Hz, 1H), 7.24-7.45 (m, 8H), 7.78-7.82 (m, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 13.5, 13.6, 13.9, 14.0, 23.0, 24.1, 24.4, 24.8, 60.6, 60.7, 60.8, 121.5, 123.4, 124.2, 124.6, 125.9, 126.4, 126.8, 127.0, 127.1, 127.2, 127.7, 127.8, 128.1, 128.2, 128.5, 129.0, 129.3, 130.0, 130.4, 130.8, 131.3, 132.3, 133.1, 134.2, 134.4, 134.6, 135.0, 136.8, 137.0, 137.3, 138.3, 141.3, 141.8, 142.2, 142.5, 143.7, 146.1, 148.0, 157.7, 158.7, 165.1, 165.3, 165.6, 165.7, 166.0, 166.8, 168.0, 168.1. HRMS Calcd (ESI) m/z for $C_{38}H_{34}NO_5Na$: $[M+H]^+$606.2256, found: 606.2253. IR ($CHCl_3$): 3410, 1716, 1578, 1519, 1444, 1220 cm^{-1}.

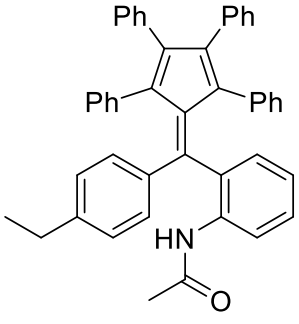


3ba

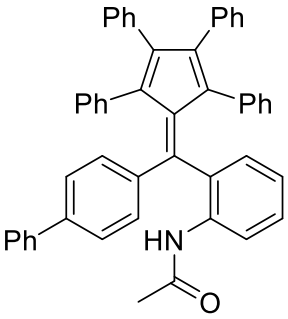
***N*-(2-((2,3,4,5-tetraphenylcyclopenta-2,4-dien-1-ylidene)(*p*-tolyl)methyl)phenyl)acetamide (3ba):** Following the general procedure **2.2**, the reaction was carried out by heating a mixture of *N*-(2-(1-(*p*-tolyl)vinyl)phenyl)acetamide **1b** (126 mg, 0.5 mmol), diphenylacetylene **2a** (178 mg, 1.0 mmol), $Pd(OAc)_2$ (11 mg, 10 mol %) and $Cu(OAc)_2.H_2O$ (150 mg, 0.75 mmol) in ^{t}AmOH (4.0 mL) at 100 ^{o}C for 18 h under air to afford the title compound in 79% yield (red solid, 239 mg). M.p.: 199–201 ^{o}C.^{1}H NMR (500 MHz, $CDCl_3$) δ 1.90 (s, 3H),

2.14 (s, 3H), 6.65-6.80 (m, 17H), 6.89 (s, 1H), 6.96-7.04 (m, 9H), 7.30 (bs, 1H), 7.69 (s, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 21.1, 24.3, 121.4, 123.2, 124.2, 125.2, 126.0, 126.1, 126.9, 127.0 , 127.8, 128.3, 129.8, 130.3, 130.5, 131.1, 132.1, 132.9, 133.0, 133.9, 134.1, 134.4, 135.3, 135.5, 136.1, 136.2, 137.0, 137.6, 139.6, 144.6, 144.9, 145.1, 151.5, 167.5. HRMS Calcd (ESI) m/z for $C_{45}H_{36}NO$: $[M+H]^+$ 606.2797, found: 606.2794. Anal. Calcd for $C_{45}H_{35}NO$: C, 89.22; H, 5.82; N, 2.31. Found: C, 89.03; H, 6.06; N, 2.46. IR ($CHCl_3$): 3406, 1686, 1577, 1517, 1442 cm^{-1}.

***N*-(2-((4-ethylphenyl)(2,3,4,5-tetraphenylcyclopenta-2,4-dien-1-ylidene)methyl)phenyl)acetamide (3ca):** Following the general procedure **2.2**, the reaction was carried out by heating a mixture of *N*-(2-(1-(4-ethylphenyl)vinyl)phenyl)acetamide **1c** (133 mg, 0.5 mmol), diphenylacetylene (**2a**, 178 mg, 1.0 mmol), $Pd(OAc)_2$ (11 mg, 10 mol %) and $Cu(OAc)_2.H_2O$ (150 mg, 0.75 mmol) in ^{t}AmOH (4.0 mL) at 100 ^{o}C for 18 h under air to afford the title compound in 54% yield (red solid, 167 mg). M.p.: 203–206 ^{o}C.^{1}H NMR (500 MHz, $CDCl_3$) δ 1.06 (t, *J* = 7.6 Hz, 3H), 1.86 (s, 3H), 2.41 (q, *J* = 7.6 Hz, 2H), 6.67–6.77 (m, 17H), 6.89 (d, *J* = 7.5 Hz, 1H), 6.94-7.04 (m, 9H), 7.24 (bs, 1H), 7.66 (d, *J* = 8.2 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 15.3, 24.3, 28.6, 121.3, 123.1, 124.5, 125.1, 126.0, 126.8, 126.9, 127.0, 127.1, 129.7, 130.3, 130.5, 131.0, 132.1, 132.7, 133.0, 133.9, 134.1, 134.3, 135.2, 135.4, 136.1, 136.9, 137.6, 144.6, 144.9, 145.0, 145.9, 151.6, 167.6. HRMS Calcd (ESI) m/z for $C_{46}H_{38}NO$: $[M+H]^+$ 620.2953, found: 620.2958. Anal. Calcd for $C_{46}H_{37}NO$: C, 89.14; H, 6.02; N, 2.26. Found: C, 89.15; H, 6.31; N, 2.55. IR ($CHCl_3$): 3401, 1598, 1578, 1519, 1443, 1220 cm^{-1}.

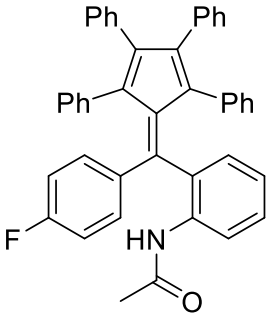


3ca



3da

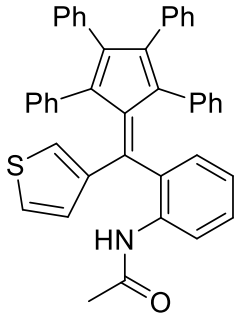
***N*-(2-([1,1'-biphenyl]-4-yl(2,3,4,5-tetraphenylcyclopenta-2,4-dien-1-ylidene)methyl)phenyl)acetamide (3da):** Following the general procedure **2.2**, the reaction was carried out by heating a mixture of *N*-(2-(1-([1,1'-biphenyl]-4-yl)vinyl)phenyl)acetamide **1d** (157 mg, 0.5 mmol), diphenylacetylene (**2a**, 178 mg, 1.0 mmol), $Pd(OAc)_2$ (11 mg, 10 mol %) and $Cu(OAc)_2.H_2O$ (150 mg, 0.75 mmol) in ^{t}AmOH (4.0 mL) at 100 ^{o}C for 18 h under air to afford the title compound in 58% yield (red solid, 194 mg). M.p.: 158–161 ^{o}C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.89 (s, 3H), 6.70-6.80 (m, 10H), 6.82 (s, 5H), 6.97-7.07 (m, 10H), 7.15 (d, *J* = 7.7 Hz, 1H), 7.31 (bs, 1H), 7.33-7.44 (m, 6H), 7.68 (d, *J* = 8.1 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 24.4, 121.7, 123.5, 124.5, 125.4, 126.0, 126.2, 126.3, 127.0, 127.1, 127.6, 128.8, 130.0, 130.4, 130.6, 131.3, 132.7, 132.9, 133.3, 134.0, 134.1, 134.4, 135.3, 135.4, 136.1, 136.2, 137.0, 139.4, 140.5, 141.9,145.2, 145.5, 150.8, 167.6. HRMS Calcd (ESI) m/z for $C_{50}H_{38}NO$: $[M+H]^+$ 668.2953, found: 668.2956. Anal. Calcd for $C_{50}H_{37}NO$: C, 89.92; H, 5.58; N, 2.10. Found: C,89.68; H, 5.50; N, 2.42. IR ($CHCl_3$): 3410, 1674, 1578, 1519, 1443, 1220 cm^{-1}.



3ea

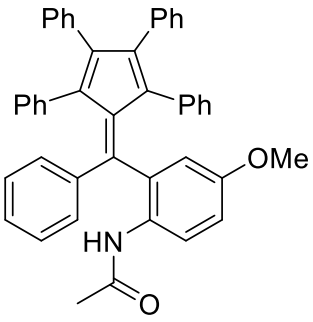
***N*-(2-((4-fluorophenyl)(2,3,4,5-tetraphenylcyclopenta-2,4-dien-1-ylidene)methyl)phenyl)acetamide (3ea):** Following the general procedure **2.2**, the reaction was carried out by heating a mixture of *N*-(2-(1-(4-fluorophenyl)vinyl)phenyl)acetamide **1e** (128 mg, 0.5 mmol), diphenylacetylene (**2a**, 178 mg, 1.0 mmol), $Pd(OAc)_2$ (11mg, 10 mol %) and $Cu(OAc)_2.H_2O$ (150 mg, 0.75 mmol) in ^{t}AmOH (4.0 mL) at 100 ^{o}C for 18 h under air to afford the title compound in 74% yield (red solid, 226 mg). M.p.: 218–220 ^{o}C.^{1}H NMR (500 MHz, $CDCl_3$) δ 1.94 (s, 3H), 6.54 (t, *J* = 8.2 Hz, 2H), 6.68-6.73 (m, 6H), 6.77-6.83 (m, 9H), 6.70-7.06 (m, 10H), 7.25 (bs, 1H), 7.58 (d, *J*

= 8.1 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 24.2, 114.2 (d, *J* = 21.4 Hz), 114.4 (d, *J* = 20.9 Hz), 115.4, 115.5, 115.6, 121.9, 123.5, 124.8, 125.3, 126.1 (d, *J* = 3.7 Hz), 127.0, 127.1, 130.0, 130.2, 130.4, 131.0, 132.5, 132.6, 133.0, 133.1, 133.6, 133.7, 134.2, 134.3, 134.4, 134.5, 135.0, 135.2, 135.8, 135.9, 136.6, 136.7, 145.0, 145.3, 145.6, 149.9, 163.1 (d, *J* = 250.1 Hz), 167.7. HRMS Calcd (ESI) m/z for $C_{44}H_{33}FNO$: $[M+H]^+$ 610.2546, found: 610.2549. Anal. Calcd for $C_{44}H_{32}FNO$: C, 86.67; H, 5.29; N, 2.30. Found: C, 86.72; H, 5.04; N, 2.12. IR ($CHCl_3$): 3410, 1674, 1598, 1504,1443, 1224 cm^{-1}.



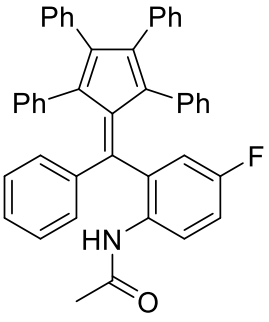
3fa

***N*-(2-((2,3,4,5-tetraphenylcyclopenta-2,4-dien-1-ylidene)(thiophen-3-yl)methyl)phenyl)acetamide (3fa):** Following the general procedure **2.2**, the reaction was carried out by heating a mixture of *N*-(2-(1-(thiophen-3-yl)vinyl)phenyl)acetamide (**1f**, 122 mg, 0.5 mmol), diphenylacetylene (**2a**, 178 mg, 1.0 mmol), $Pd(OAc)_2$ (11 mg, 10 mol %) and $Cu(OAc)_2.H_2O$ (150 mg, 0.75 mmol) in ^{t}AmOH (4.0 mL) at 100 °C for 18 h under air to afford the title compound in 59% yield (brown solid, 176 mg). M.p.: 200-202 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.91 (s, 3H), 6.64 (d, *J* = 4.7 Hz, 1H), 6.67-6.72 (m, 5H), 6.77 (s, 4H), 6.80-6.87 (m, 6H), 6.94-7.05 (m, 10H), 7.23 (s, 1H), 7.66 (d, *J* = 8.1 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 24.3, 121.5, 122.2, 123.2, 124.7, 124.9, 125.2, 126.1, 126.4, 126.9, 127.0, 127.1, 128.5, 129.9, 130.1, 130.2, 130.4, 130.7, 131.0, 132.2, 132.4, 133.5, 133.9, 134.2, 134.5, 135.1, 135.4, 136.0, 136.1, 136.9, 141.9, 144.6, 144.8, 145.1, 167.6. HRMS Calcd (ESI) m/z for $C_{42}H_{32}NOS$: $[M+H]^+$ 598.2205, found: 598.2209. Anal. Calcd for $C_{42}H_{31}NOS$: C, 84.39; H, 5.23; N, 2.34. Found: C, 84.63; H, 5.20; N, 2.08. IR ($CHCl_3$): 3401, 1664, 1220 cm^{-1}.



3ha

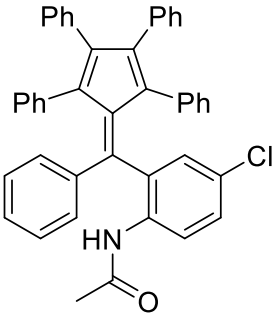
***N*-(5-methoxy-2-(phenyl(2,3,4,5-tetraphenylcyclopenta-2,4-dien-1-ylidene)methyl)phenyl)acetamide (3ha):** Following the general procedure **2.2**, the reaction was carried out by heating a mixture of *N*-(5-methoxy-2-(1-phenylvinyl)phenyl)acetamide **1h** (134 mg, 0.5 mmol), diphenylacetylene (**2a**, 178 mg, 1.0 mmol), $Pd(OAc)_2$ (11 mg, 10 mol %) and $Cu(OAc)_2.H_2O$ (150 mg, 0.75 mmol) in ^{t}AmOH (4.0 mL) at 100 ^{o}C for 18 h under air to afford the title compound in 61% yield (brown solid, 190 mg). M.p.: 152–155 ^{o}C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.82 (s, 3H), 3.63 (s, 3H), 6.52 (dd, *J* = 8.9, 2.7 Hz, 1H), 6.57 (d, *J* = 2.3 Hz, 1H), 6.70-6.72 (m, 3H), 6.75 (s, 5H), 6.83-6.87 (m, 7H), 6.93-7.05 (m, 10H), 7.18 (d, *J* = 6.8 Hz, 1H), 7.44 (d, *J* = 8.9 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 24.0, 55.5, 115.4, 119.2, 124.4, 124.8, 125.3, 126.2, 127.0, 127.1, 127.3, 127.7, 128.4, 129.2, 129.7, 130.0, 130.4, 130.6, 131.1, 131.7, 133.2, 134.2, 134.6, 135.0, 135.3, 135.5, 136.4, 137.0, 140.2, 145.1, 145.4, 145.5, 151.4, 155.2, 167.7. HRMS Calcd (ESI) m/z for $C_{45}H_{36}NO_2$: $[M+H]^+$ 622.2746, found: 622.2748. Anal. Calcd for $C_{45}H_{35}NO_2$: C, C, 86.93; H, 5.67; N, 2.25. Found: C, 86.68; H, 5.60; N, 1.97. IR ($CHCl_3$): 3410, 1665, 1601, 1514, 1220 cm^{-1}.



3ia

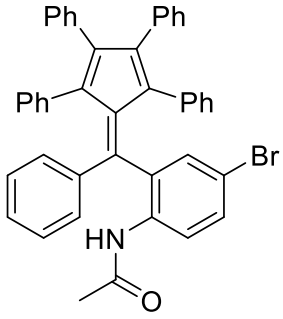
***N*-(5-fluoro-2-(phenyl(2,3,4,5-tetraphenylcyclopenta-2,4-dien-1-ylidene)methyl)phenyl)acetamide (3ia):** Following the general procedure **2.2**, the reaction was carried out by heating a mixture of *N*-(5-fluoro-2-(1-phenylvinyl)phenyl)acetamide (**1i**, 128 mg, 0.5 mmol), diphenylacetylene (**2a**, 178 mg, 1.0 mmol), $Pd(OAc)_2$ (11 mg, 10 mol %) and $Cu(OAc)_2.H_2O$ (150 mg, 0.75 mmol) in ^{t}AmOH (4.0 mL) at 100 ^{o}C for 18 h under air to afford the title compound in 76% yield (red solid, 232 mg). M.p.: 205-208 ^{o}C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.86 (s, 3H), 6.64-6.68 (m, 1H), 6.71 (d, *J* = 7.2 Hz, 4H), 6.74-6.76 (m, 6H),

6.83-6.87 (m, 5H), 6.92-7.06 (m, 11H), 7.15 (s, 1H), 7.57 (d, J = 8.9 Hz, 1H).^{13}C NMR (125 MHz, $CDCl_3$) δ 24.1, 116.2 (d, J = 21.9 Hz), 116.3, 120.4 (d, J = 22.9 Hz), 124.0, 124.9, 125.6, 126.3, 126.4, 127.1, 127.3, 127.5, 127.8, 128.6, 129.4, 130.0, 130.3, 130.5, 131.1, 131.8, 132.3, 133.0, 134.1, 134.2, 134.9 (d, J = 7.5 Hz), 135.0, 135.2, 136.2, 136.7, 139.8, 145.8, 146.0, 149.4, 158.3 (d, J = 254.0), 167.7. HRMS Calcd (ESI) m/z for $C_{44}H_{33}FNO$: $[M+H]^+$ 610.2546, found: 610.2549. Anal. Calcd for $C_{44}H_{32}FNO$:C, 86.67; H, 5.29; N, 2.30. Found:C, 86.88; H, 5.40; N, 1.98. IR ($CHCl_3$): 3410, 1676, 1600, 1517, 1228 cm^{-1}.



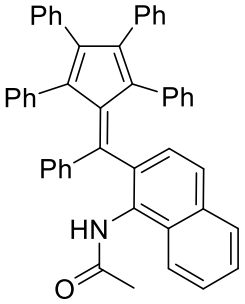
3ja

***N*-(4-chloro-2-(phenyl(2,3,4,5-tetraphenylcyclopenta-2,4-dien-1-ylidene)methyl)phenyl)acetamide (3ja):** Following the general procedure **2.2**, the reaction was carried out by heating a mixture of *N*-(5-chloro-2-(1-phenylvinyl)phenyl)acetamide **1j** (136 mg, 0.5 mmol), diphenylacetylene (**2a**, 178 mg, 1.0 mmol), $Pd(OAc)_2$ (11 mg, 10 mol %) and $Cu(OAc)_2.H_2O$ (150 mg, 0.75 mmol) in ^{t}AmOH (4.0 mL) at 100 ^{o}C for 18 h under air to afford the title compound in 77% yield (red solid, 241 mg). M.p.: 212–215 ^{o}C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.80 (s, 3H), 6.71 (t, J = 6.8 Hz, 4H), 6.76-6.78 (m, 5H), 6.83-6.92 (m, 7H), 6.95-7.07 (m, 11H), 7.18 (d, J = 7.5 Hz, 1H), 7.66 (d, J = 8.8 Hz, 1H).^{13}C NMR (125 MHz, $CDCl_3$) δ 24.3, 122.9, 125.0, 125.6, 126.4, 127.1, 127.3, 127.5, 128.0, 128.4, 128.7, 129.4, 129.5, 130.0, 130.3, 130.5, 131.1, 131.3, 133.1, 133.2, 133.7, 133.9, 134.5, 134.9, 135.0, 135.2, 136.4, 136.7, 139.5, 145.8, 146.0, 146.2, 149.2, 167.6. HRMS Calcd (ESI) m/z for $C_{44}H_{33}ClNO$: $[M+H]^+$ 626.2251, found: 626.2247. Anal. Calcd for $C_{44}H_{32}ClNO$: C, 84.40; H, 5.15; N, 2.24. Found: C, 84.17; H, 5.37; N, 2.03. IR ($CHCl_3$): 3400, 1660, 1591, 1220 cm^{-1}.



3ka

***N*-(5-bromo-2-(phenyl(2,3,4,5-tetraphenylcyclopenta-2,4-dien-1-ylidene)methyl)phenyl)acetamide (3ka):** Following the general procedure **2.2**, the reaction was carried out by heating a mixture of *N*-(5-bromo-2-(1-phenylvinyl)phenyl)acetamide **1k** (158 mg, 0.5 mmol), diphenylacetylene (**2a**, 178 mg, 1.0 mmol), $Pd(OAc)_2$ (11 mg, 10 mol %) and $Cu(OAc)_2.H_2O$ (150 mg, 0.75 mmol) in ^{t}AmOH (4.0 mL) at 100 ^{o}C for 18 h under air to afford the title compound in 68% yield (red solid, 228 mg). M.p.: 209–212 ^{o}C.^{1}H NMR (500 MHz, $CDCl_3$) δ 1.78 (s, 3H), 6.70 (t, *J* = 6.8 Hz, 4H), 6.73-6.77 (m, 5H), 6.82 (t, *J* = 6.8 Hz, 4H), 6.89 (bs, 1H), 6.95-7.06 (m, 12H), 7.16–7.19 (m, 2H), 7.61 (d, *J* = 8.8 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 24.3, 116.3, 123.0, 125.0, 125.6, 126.4, 127.1, 127.3, 127.5, 128.0, 128.6, 129.4, 130.0, 130.3, 130.5, 131.1, 131.3, 132.3, 133.1, 133.8, 133.9, 134.5, 135.0, 135.2, 135.4, 135.5, 136.4, 136.7, 139.4, 145.8, 146.0, 146.3, 149.0, 167.5. HRMS Calcd (ESI) m/z for $C_{44}H_{33}BrNO$: $[M+H]^+$ 670.1746, found: 670.1744. Anal. Calcd for $C_{44}H_{32}BrNO$: C, 78.80; H, 4.81; N, 2.09. Found: C, 78.84; H, 4.80; N, 2.37. IR ($CHCl_3$): 3408, 1672, 1600, 1504, 1220 cm^{-1}.

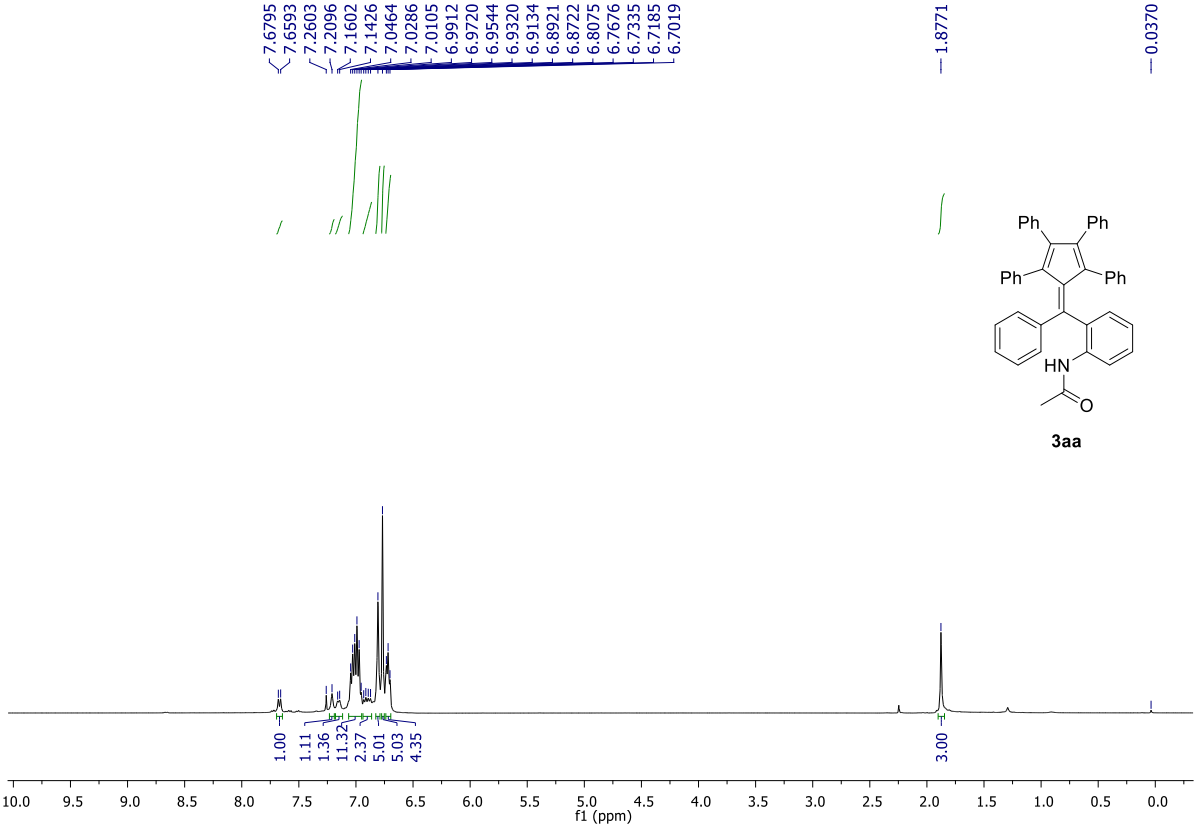


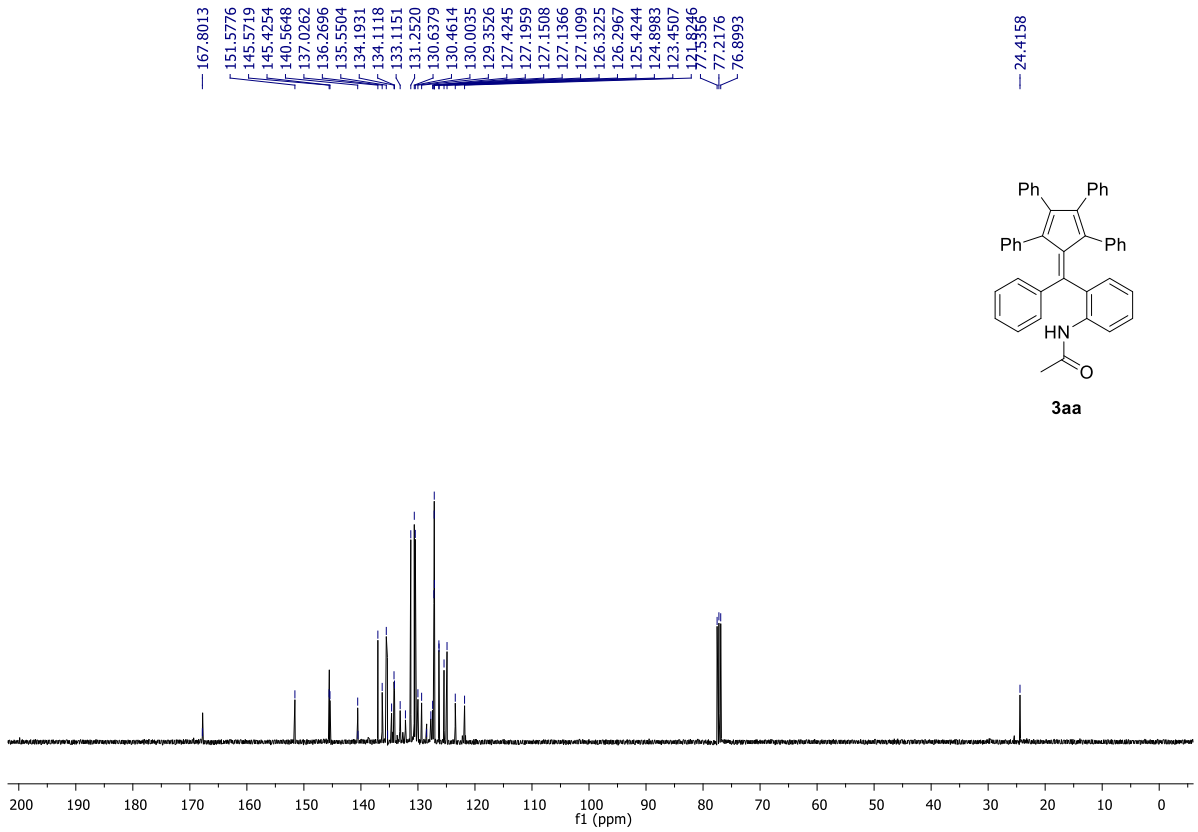
3la

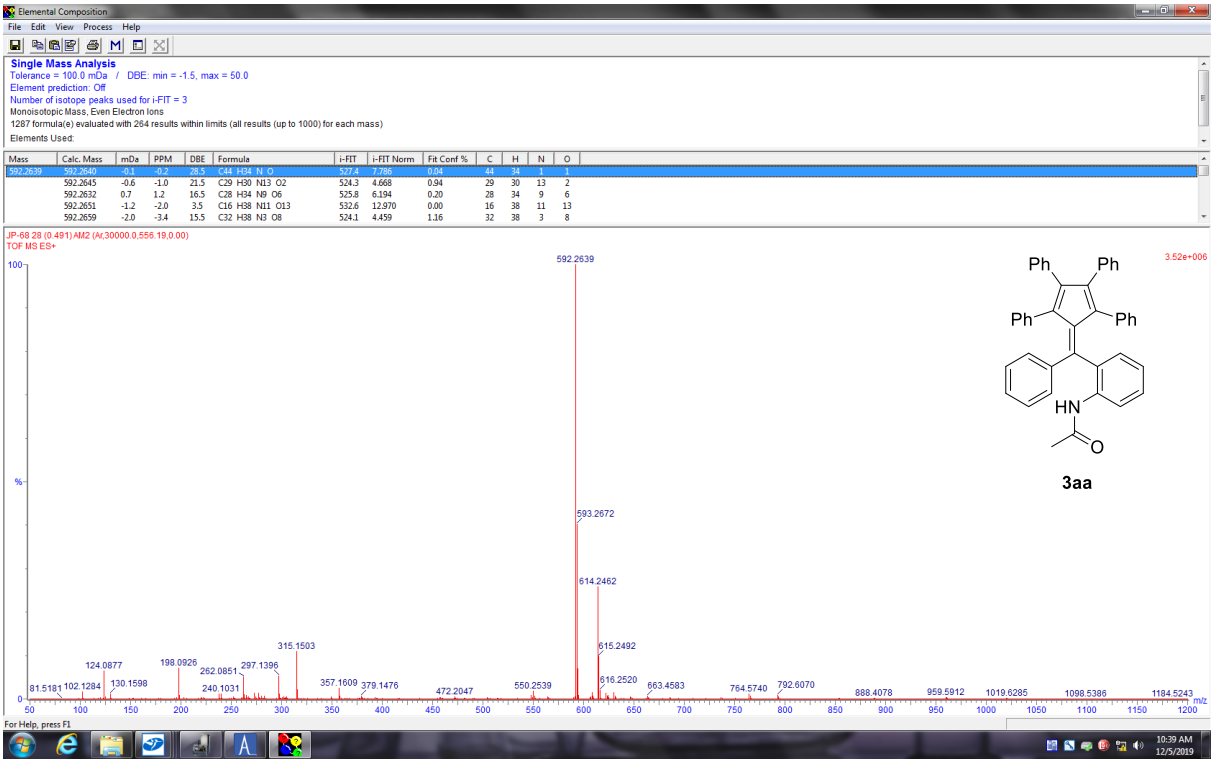
***N*-(2-(phenyl(2,3,4,5-tetraphenylcyclopenta-2,4-dien-1-ylidene)methyl)naphthalen-1-yl)acetamide (3la):** Following the general procedure **2.2**, the reaction was carried out by heating a mixture of *N*-(2-(1-phenylvinyl)naphthalen-1-yl)acetamide **1l** (144 mg, 0.5 mmol), diphenylacetylene (**2a**, 178 mg, 1.0 mmol), $Pd(OAc)_2$ (11 mg, 10 mol %) and $Cu(OAc)_2.H_2O$ (150 mg, 0.75 mmol) in ^{t}AmOH (4.0 mL) at 100 ^{o}C for 18 h under air to afford the title compound in 70% yield (red solid, 225 mg). M.p.: 188–191 ^{o}C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.24 (s, 3H), 6.31 (t, *J* = 9.4 Hz, 1H), 6.45-6.52 (m, 2H), 6.68-6.86 (m, 12H), 6.93-7.04 (m, 9H), 7.71 (s, 1H), 7.14-7.22

(m, 2H), 7.34-7.45 (m, 3H), 7.57-7.70 (m, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 22.9, 123.9, 124.6, 124.8, 125.0, 126.2, 126.4, 126.9, 127.0, 127.2, 127.3, 127.4, 127.7, 129.0, 129.7, 130.0, 130.3, 130.5, 130.7, 131.2, 132.8, 133.9, 134.2, 134.3, 135.0, 135.4, 135.6, 135.9, 137.1, 138.3, 140.9, 140.9, 144.4, 144.7, 146.0, 153.0, 169.7. HRMS Calcd (ESI) m/z for $C_{48}H_{36}NO$: $[M+H]^+$ 642.2797, found: 642.2794. Anal. Calcd for $C_{48}H_{35}NO$: C, 89.83; H, 5.50; N, 2.18. Found: C, 90.16; H, 5.42; N, 1.92. IR ($CHCl_3$): 3401, 2344, 1680, 1485, 1442, 1249 cm^{-1}.

7. NMR-spectra



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Single Mass Analysis
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Element prediction: Off
Number of isotope peaks used for i-FIT = 3
Monoisotopic Mass, Even Electron Ions
1287 formula(e) evaluated with 264 results within limits (all results (up to 1000) for each mass)
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592.2632 0.7 1.2 16.5 C28 H34 N9 O6 525.8 6.194 0.20 28 34 9 6
592.2651 -1.2 -2.0 3.5 C16 H38 N11 O13 532.6 12.970 0.00 16 38 11 13
592.2659 -2.0 -3.4 15.5 C32 H38 N3 O8 524.1 4.459 1.16 32 38 3 8
JP-68 28 (0.491) AM2 (Ar,30000.0,556.19,0.00)
TOF MS ES+
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616.2520
315.1503
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198.0926
262.0851
297.1396
240.1031
130.1598
102.1284
81.5181
357.1609
379.1476
472.2047
550.2539
663.4583
764.5740
792.6070
888.4078
959.5912
1019.6285
1098.5386
1184.5243
m/z
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Ph
Ph
Ph
Ph
HN
O
3aa
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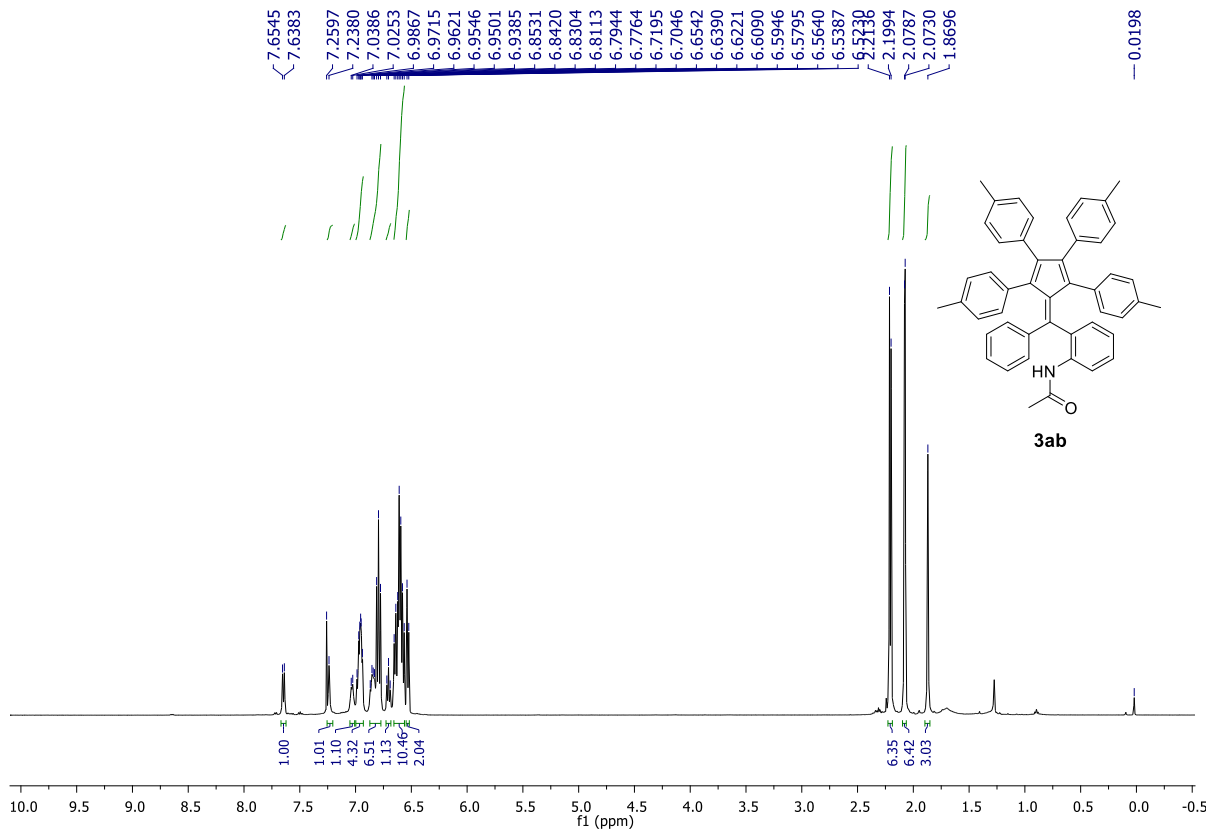
10:39 AM
12/5/2019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6.9501
6.9385
6.8531
6.8420
6.8304
6.8113
6.7944
6.7764
6.7195
6.7046
6.6542
6.6390
6.6221
6.6090
6.5946
6.5795
6.5640
6.5387
2.1994
2.0787
2.0730
1.8696
0.0198
HN
O
3ab
1.00
1.01
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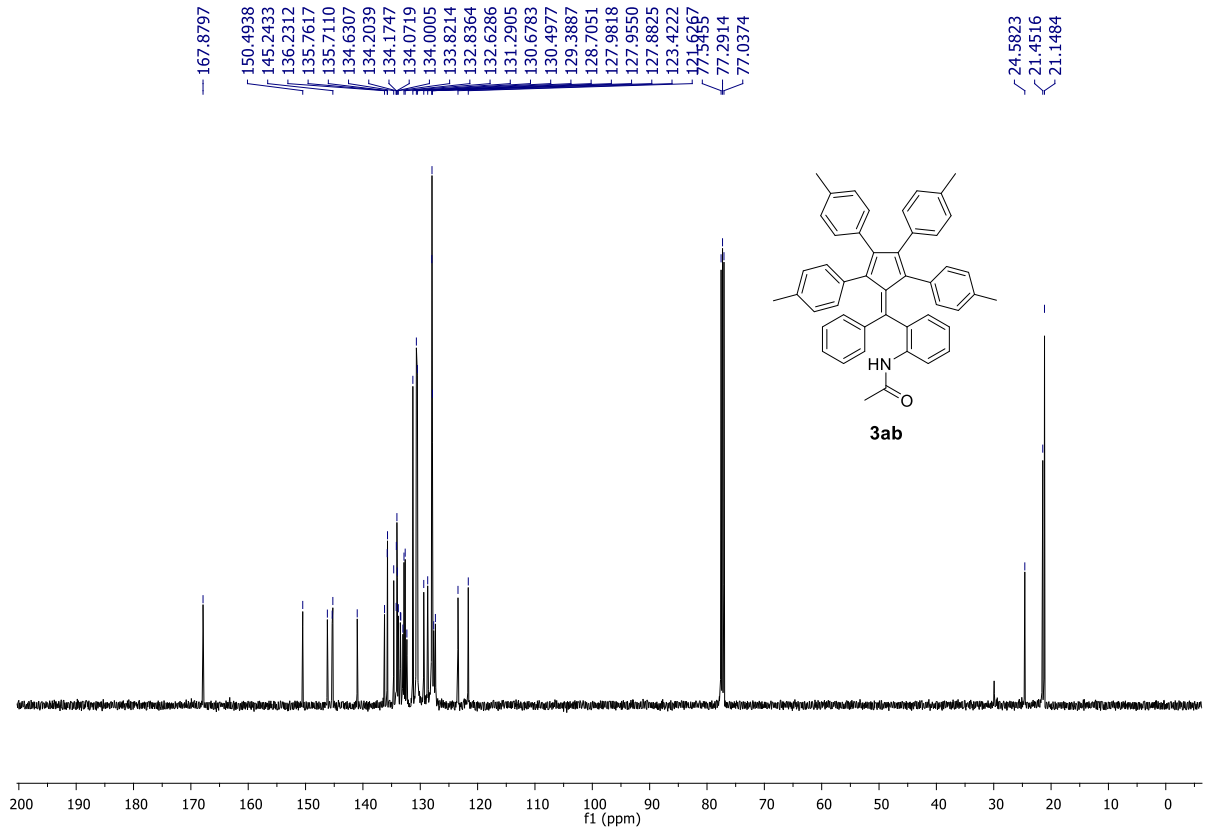
4.32
6.51
1.13
10.46
2.04
6.35
6.42
3.03
f1 (ppm)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134.0005
133.8214
132.8364
132.6286
131.2905
130.6783
130.4977
129.3887
128.7051
127.9818
127.9550
127.8825
123.4222
121.6267
77.3455
77.2914
77.0374
24.5823
21.4516
21.1484
HN
O
3ab
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190
180
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110
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50
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10
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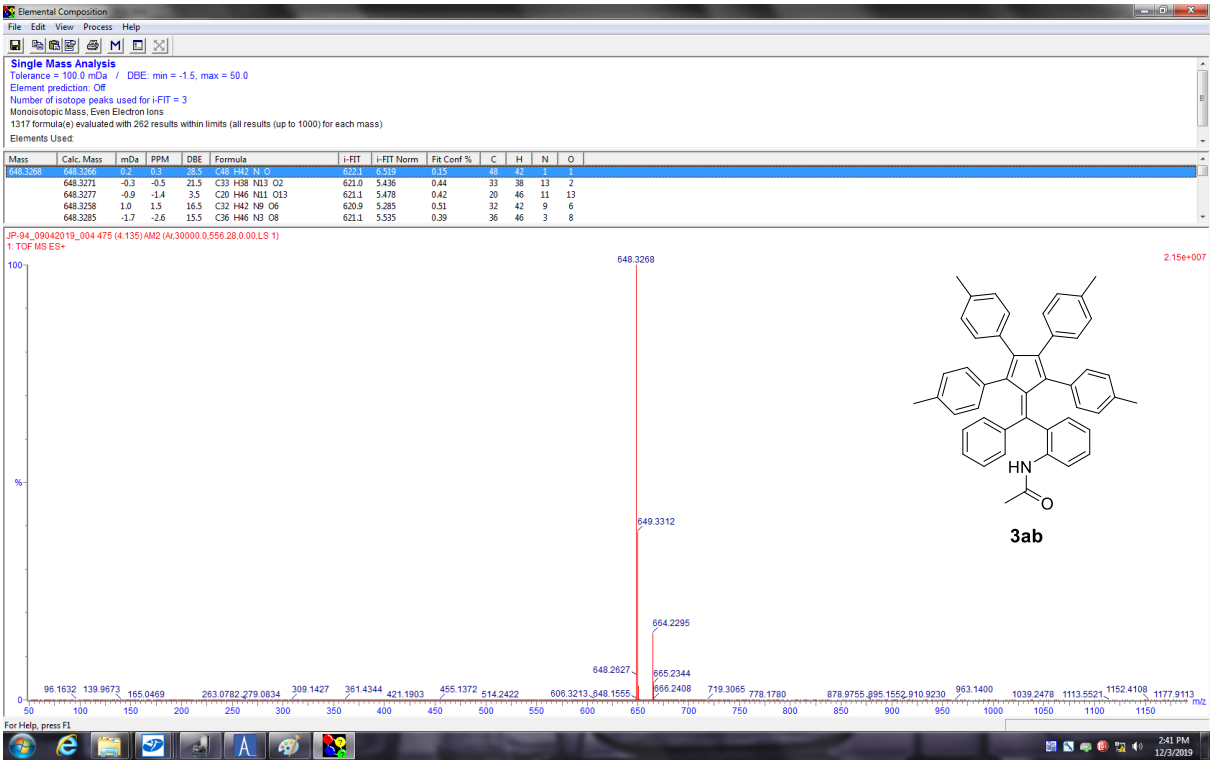
f1 (ppm)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File Edit View Process Help
Single Mass Analysis
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Element prediction: Off
Number of isotope peaks used for i-FIT = 3
Monoisotopic Mass, Even Electron Ions
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648.3277 -0.9 -1.4 3.5 C20 H46 N11 O13 621.1 5.478 0.42 20 46 11 13
648.3258 1.0 1.5 16.5 C32 H42 N9 O6 620.9 5.285 0.51 32 42 9 6
648.3285 -1.7 -2.6 15.5 C36 H46 N3 O8 621.1 5.535 0.39 36 46 3 8
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%
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666.2408
719.3065
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963.1400
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1152.4108
1177.9113
m/z
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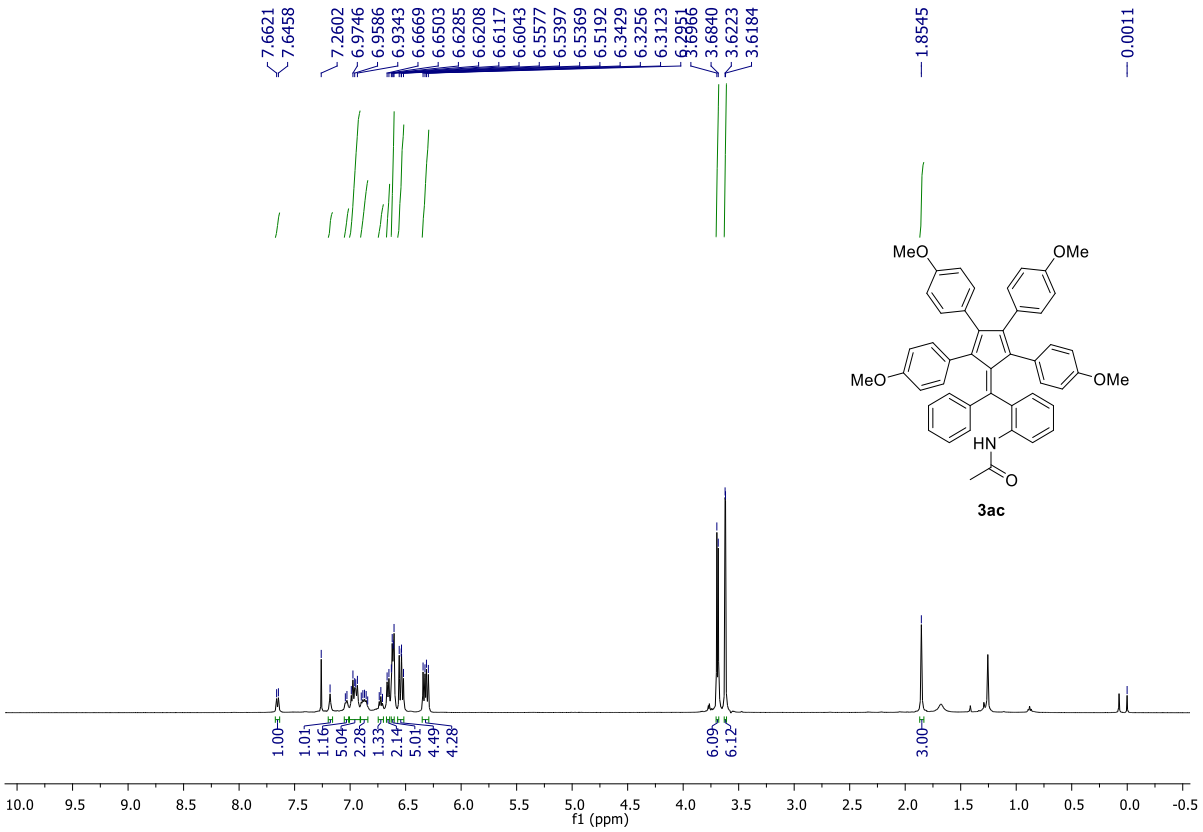
HN
O
3ab
For Help, press F1
2:41 PM
12/3/2019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6.5192
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6.3256
6.3123
6.2951
3.6966
3.6840
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3.6184
1.8545
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MeO
OMe
MeO
OMe
HN
O
3ac
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5.01
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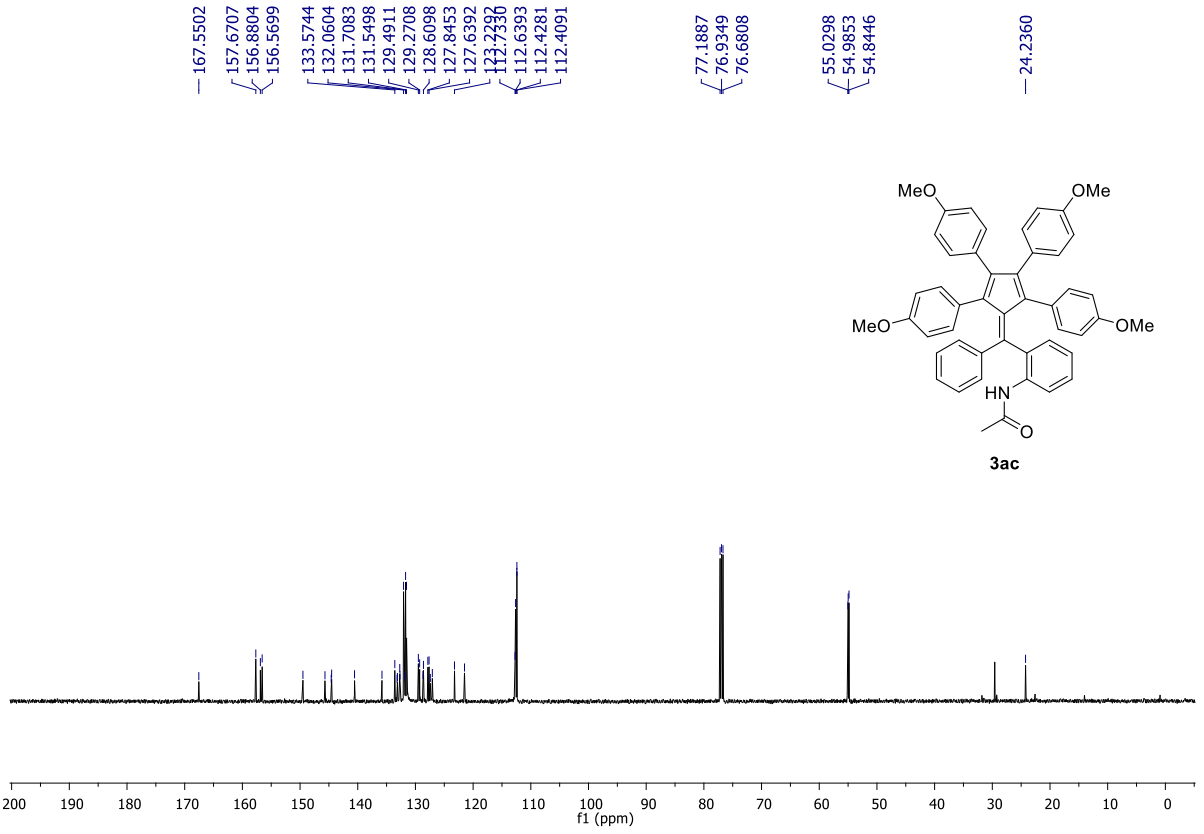
f1 (ppm)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127.8453
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76.9349
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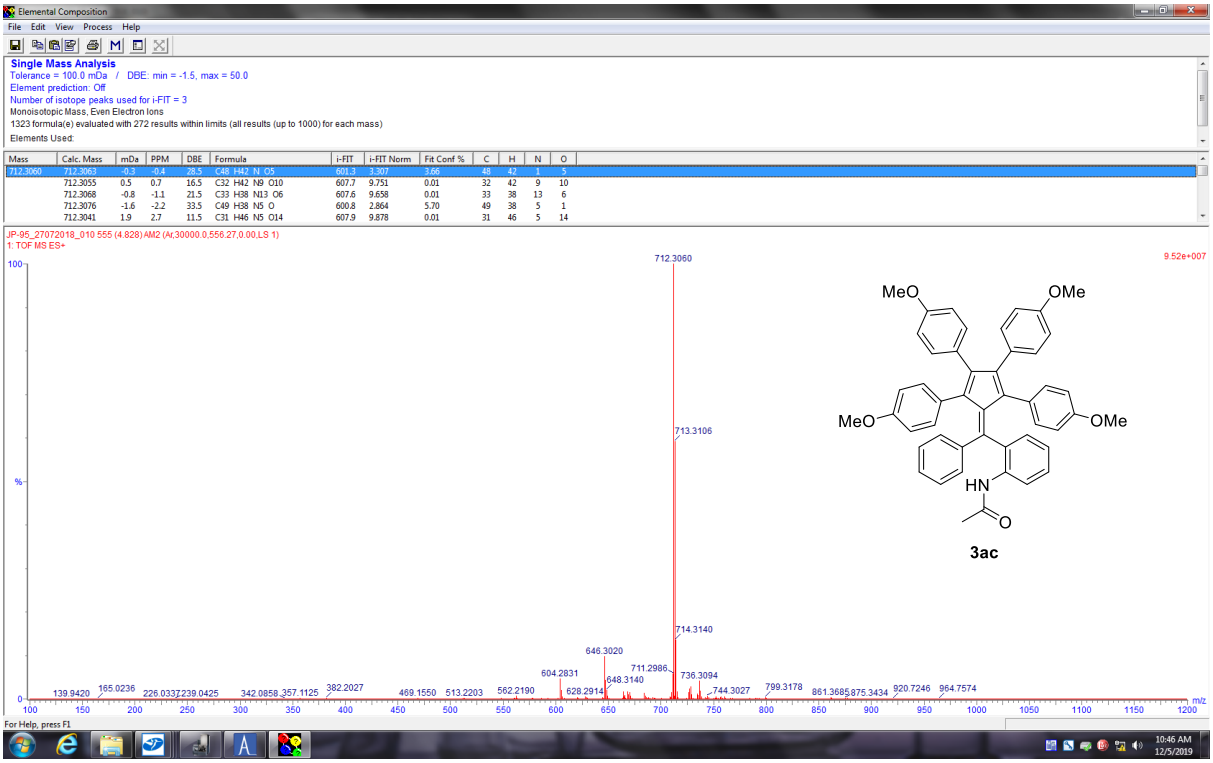
O
3ac
f1 (ppm)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Monoisotopic Mass, Even Electron Ions
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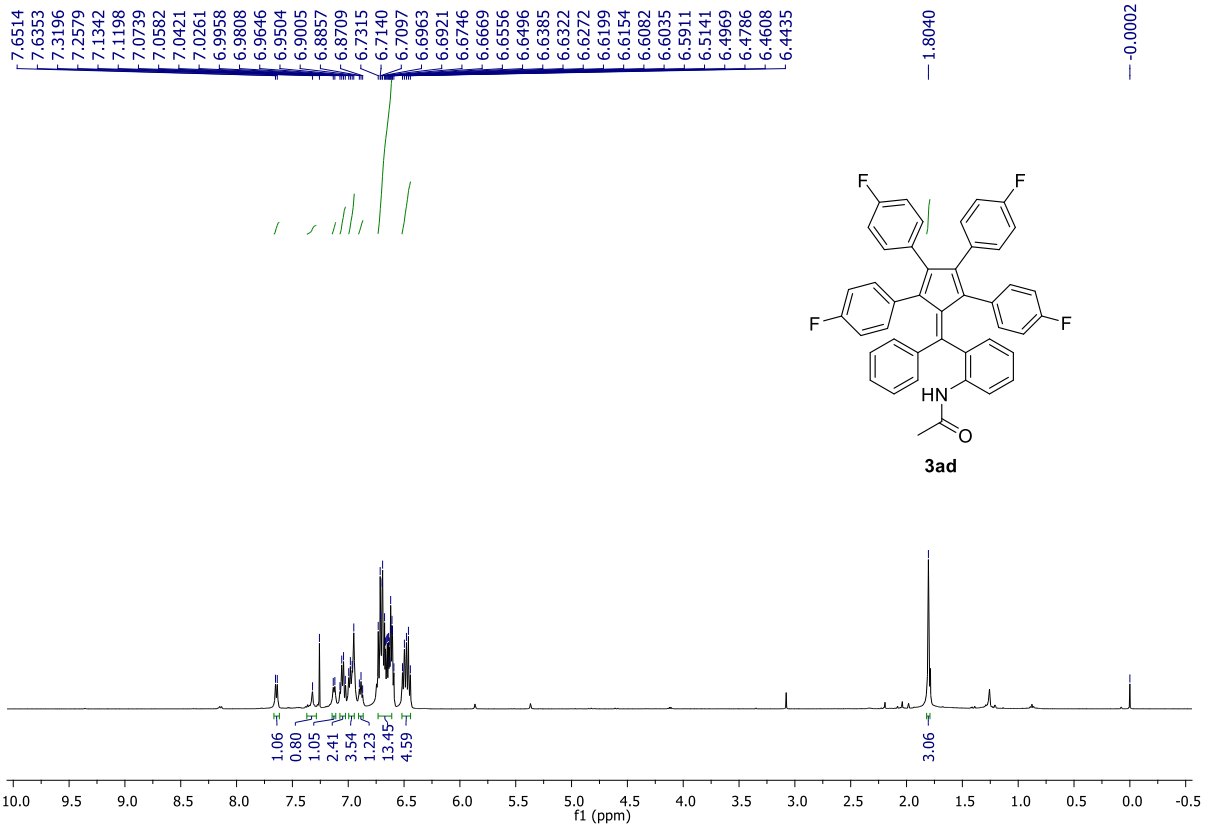
O
3ac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6.9808
6.9646
6.9504
6.9005
6.8857
6.8709
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6.7140
6.7097
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6.6746
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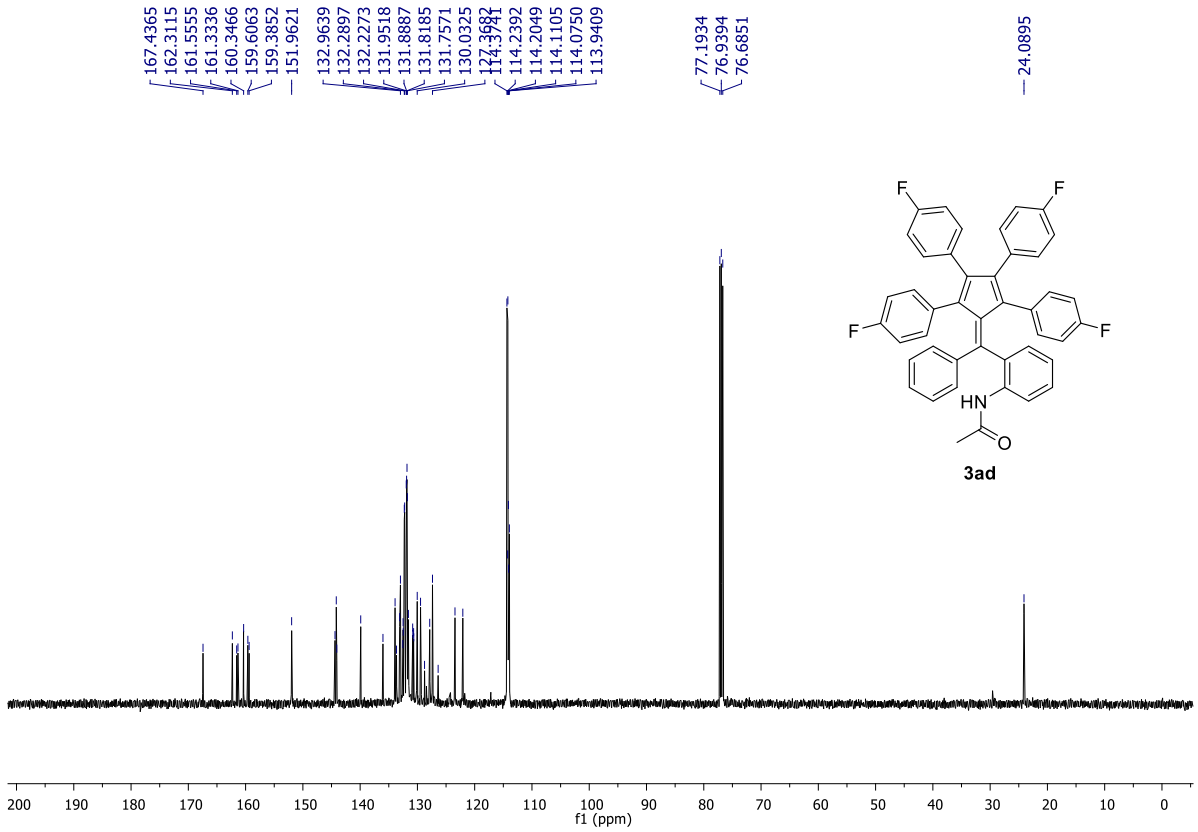
f1 (ppm)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F
F
HN
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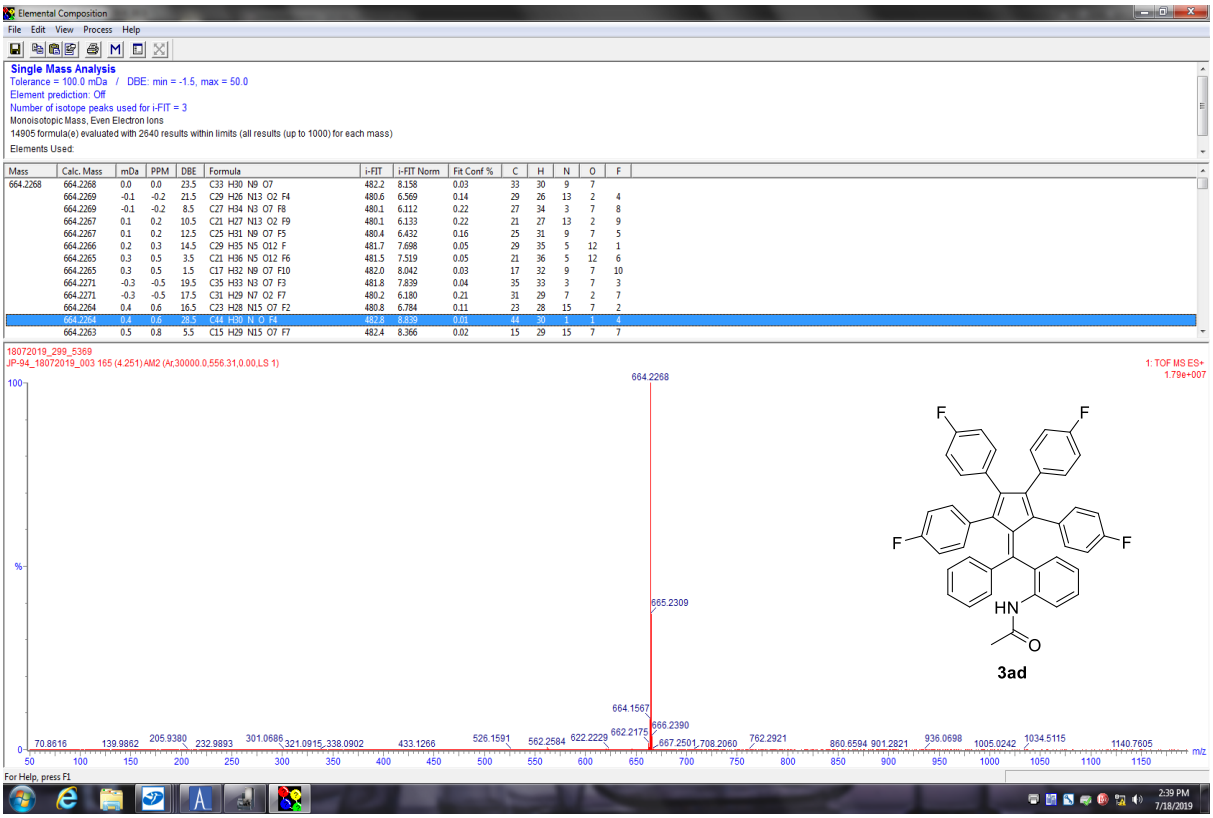
f1 (ppm)


Elemental Composition
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Single Mass Analysis
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Monoisotopic Mass, Even Electron Ions
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664.2269 | -0.1 | -0.2 | 21.5 | C29 H26 N13 O2 F4 | 480.6 | 6.569 | 0.14 | 29 | 26 | 13 | 2 | 4
664.2269 | -0.1 | -0.2 | 8.5 | C27 H34 N3 O7 F8 | 480.1 | 6.112 | 0.22 | 27 | 34 | 3 | 7 | 8
664.2267 | 0.1 | 0.2 | 10.5 | C21 H27 N13 O2 F9 | 480.1 | 6.133 | 0.22 | 21 | 27 | 13 | 2 | 9
664.2267 | 0.1 | 0.2 | 12.5 | C25 H31 N9 O7 F5 | 480.4 | 6.432 | 0.16 | 25 | 31 | 9 | 7 | 5
664.2266 | 0.2 | 0.3 | 14.5 | C29 H35 N5 O12 F | 481.7 | 7.698 | 0.05 | 29 | 35 | 5 | 12 | 1
664.2265 | 0.3 | 0.5 | 3.5 | C21 H36 N5 O12 F6 | 481.5 | 7.519 | 0.05 | 21 | 36 | 5 | 12 | 6
664.2265 | 0.3 | 0.5 | 1.5 | C17 H32 N9 O7 F10 | 482.0 | 8.042 | 0.03 | 17 | 32 | 9 | 7 | 10
664.2271 | -0.3 | -0.5 | 19.5 | C35 H33 N3 O7 F3 | 481.8 | 7.839 | 0.04 | 35 | 33 | 3 | 7 | 3
664.2271 | -0.3 | -0.5 | 17.5 | C31 H29 N7 O2 F7 | 480.2 | 6.180 | 0.21 | 31 | 29 | 7 | 2 | 7
664.2264 | 0.4 | 0.6 | 16.5 | C23 H28 N15 O7 F2 | 480.8 | 6.784 | 0.11 | 23 | 28 | 15 | 7 | 2
664.2264 | 0.4 | 0.6 | 28.5 | C44 H30 N O F4 | 482.8 | 8.839 | 0.01 | 44 | 30 | 1 | 1 | 4
664.2263 | 0.5 | 0.8 | 5.5 | C15 H29 N15 O7 F7 | 482.4 | 8.366 | 0.02 | 15 | 29 | 15 | 7 | 7
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m/z
50
100
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200
250
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350
400
450
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550
600
650
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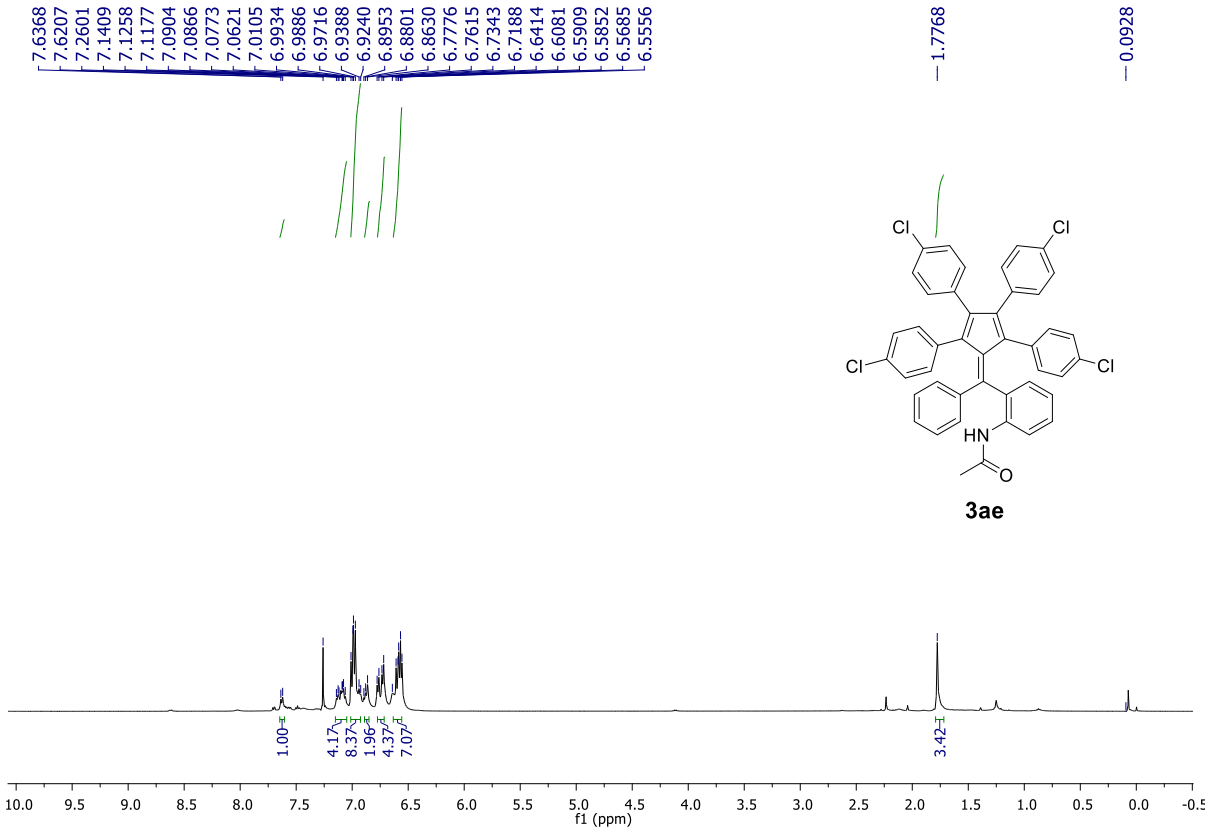
7/18/2019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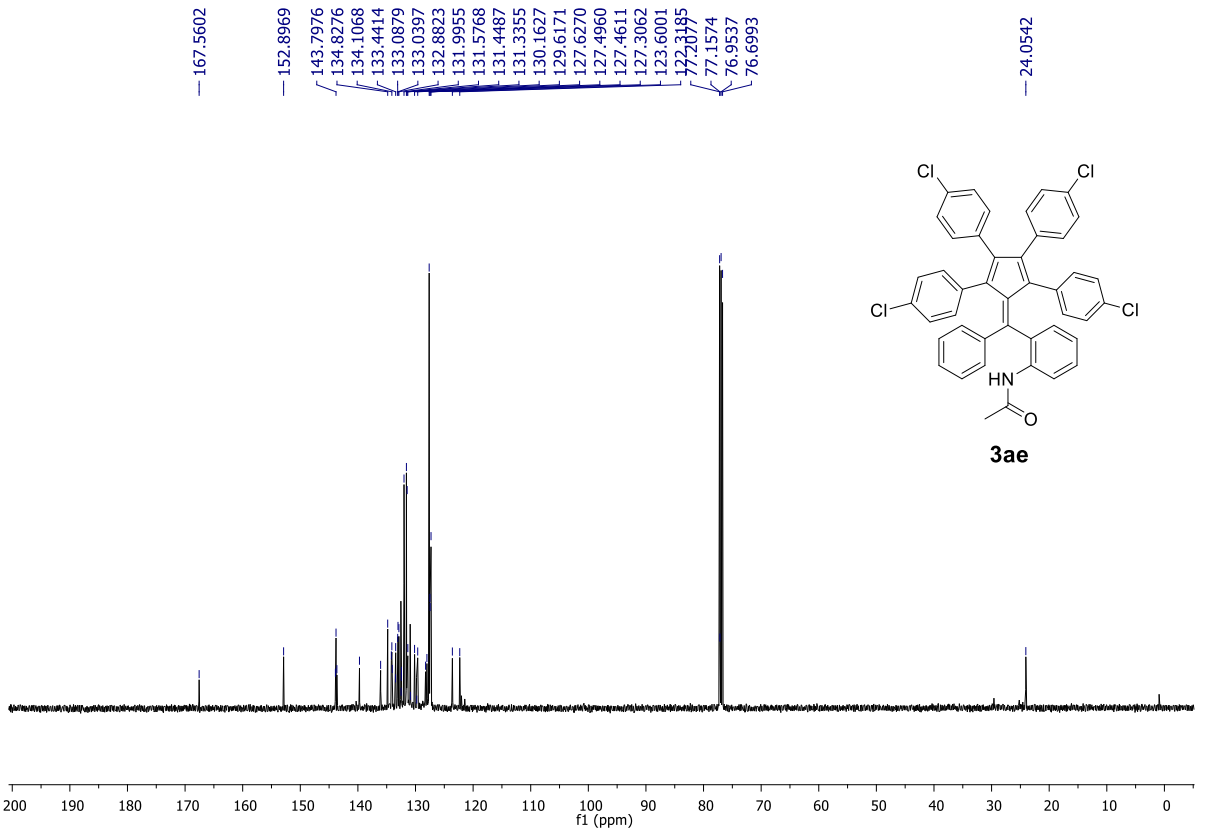
f1 (ppm)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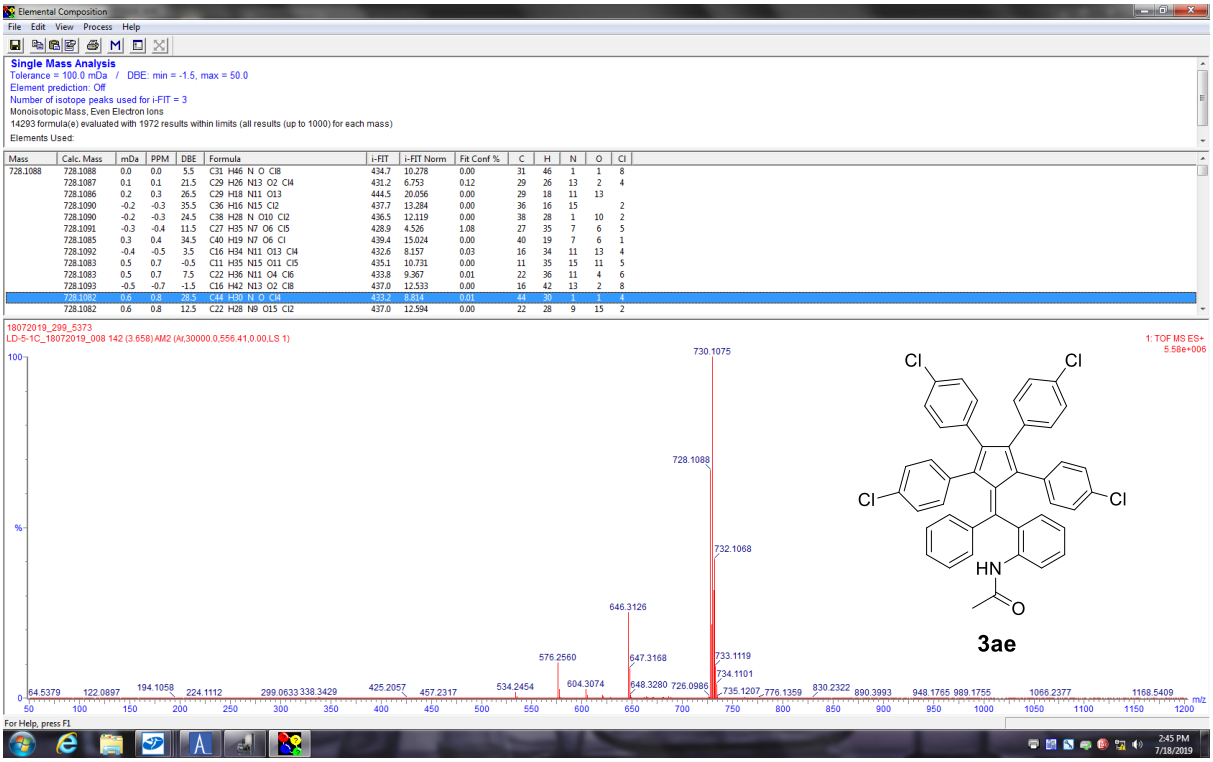
3ae
f1 (ppm)


Elemental Composition
Single Mass Analysis
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Element prediction: Off
Number of isotope peaks used for i-FIT = 3
Monoisotopic Mass, Even Electron Ions
14293 formula(e) evaluated with 1972 results within limits (all results (up to 1000) for each mass)
Elements Used:
Mass Calc. Mass mDa PPM DBE Formula i-FIT i-FIT Norm Fit Conf % C H N O Cl
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728.1086 0.2 0.3 26.5 C29 H18 N11 O13 444.5 20.056 0.00 29 18 11 13
728.1090 -0.2 -0.3 35.5 C36 H16 N15 Cl2 437.7 13.284 0.00 36 16 15 2
728.1090 -0.2 -0.3 24.5 C38 H28 N O10 Cl2 436.5 12.119 0.00 38 28 1 10 2
728.1091 -0.3 -0.4 11.5 C27 H35 N7 O6 Cl5 428.9 4.526 1.08 27 35 7 6 5
728.1085 0.3 0.4 34.5 C40 H19 N7 O6 Cl 439.4 15.024 0.00 40 19 7 6 1
728.1092 -0.4 -0.5 3.5 C16 H34 N11 O13 Cl4 432.6 8.157 0.03 16 34 11 13 4
728.1083 0.5 0.7 -0.5 C11 H35 N15 O11 Cl5 435.1 10.731 0.00 11 35 15 11 5
728.1083 0.5 0.7 7.5 C22 H36 N11 O4 Cl6 433.8 9.367 0.01 22 36 11 4 6
728.1093 -0.5 -0.7 -1.5 C16 H42 N13 O2 Cl8 437.0 12.533 0.00 16 42 13 2 8
728.1082 0.6 0.8 28.5 C44 H30 N O Cl4 433.2 8.814 0.01 44 30 1 1 4
728.1082 0.6 0.8 12.5 C22 H28 N9 O15 Cl2 437.0 12.594 0.00 22 28 9 15 2
18072019_299_5373
LD-5-1C_18072019_008 142 (3.658) AM2 (Ar,30000.0,556.41,0.00,LS 1)
1: TOF MS ES+
5.58e+006
730.1075
728.1088
732.1068
646.3126
576.2560
647.3168
733.1119
734.1101
604.3074
648.3280
726.0986
735.1207
776.1359
830.2322
890.3993
948.1765
989.1755
1066.2377
1168.5409
64.5379
122.0897
194.1058
224.1112
299.0633
338.3429
425.2057
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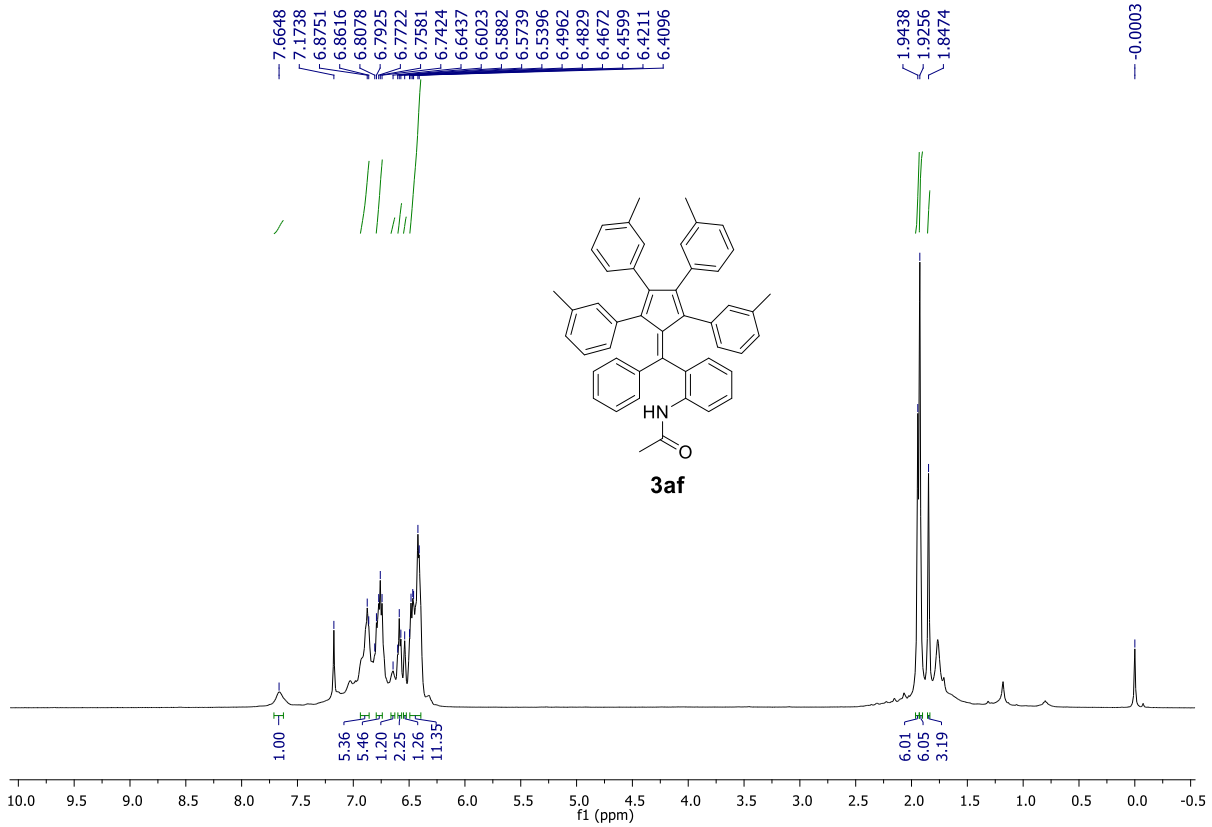
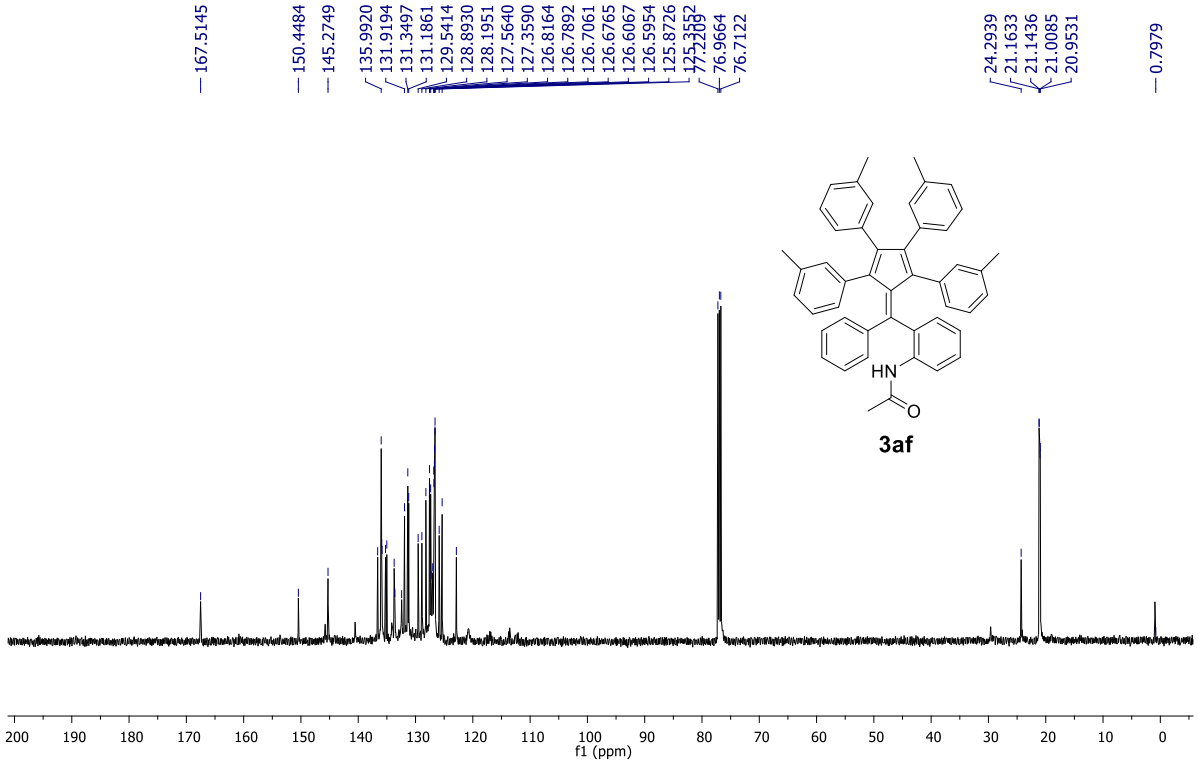
Cl
Cl
Cl
Cl
HN
O
3ae
For Help, press F1
2:45 PM
7/18/2019


7.6648
7.1738
6.8751
6.8616
6.8078
6.7925
6.7722
6.7581
6.7424
6.6437
6.6023
6.5882
6.5739
6.5396
6.4962
6.4829
6.4672
6.4599
6.4211
6.4096
1.9438
1.9256
1.8474
-0.0003
HN
O
3af
1.00
5.36
5.46
1.20
2.25
1.26
11.35
6.01
6.05
3.19
f1 (ppm)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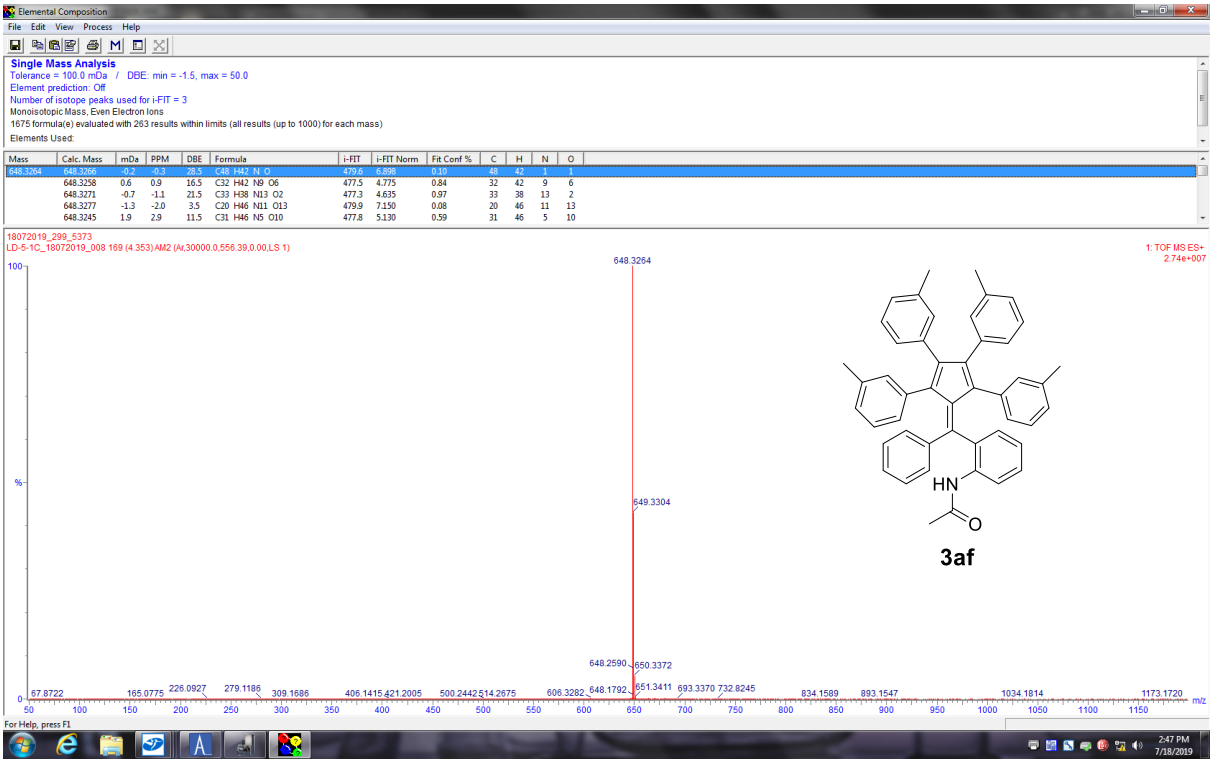
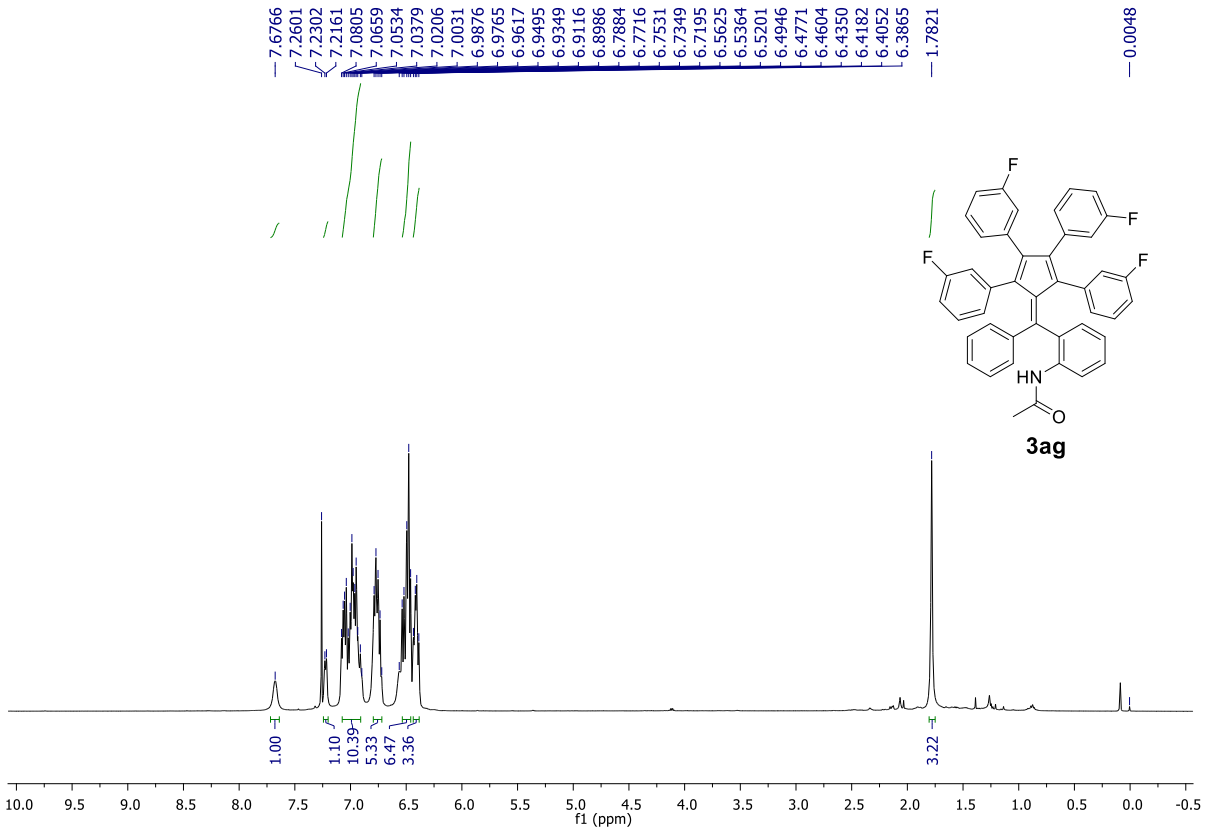
145.2749
135.9920
131.9194
131.3497
131.1861
129.5414
128.8930
128.1951
127.5640
127.3590
126.8164
126.7892
126.7061
126.6765
126.6067
126.5954
125.8726
77.2209
76.9664
76.7122
24.2939
21.1633
21.1436
21.0085
20.9531
0.7979
HN
O
3af
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


Elemental Composition
File Edit View Process Help
Single Mass Analysis
Tolerance = 100.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3
Monoisotopic Mass, Even Electron Ions
1675 formula(e) evaluated with 263 results within limits (all results (up to 1000) for each mass)
Elements Used:
Mass Calc. Mass mDa PPM DBE Formula i-FIT i-FIT Norm Fit Conf % C H N O
648.3264 648.3266 -0.2 -0.3 28.5 C48 H42 N O 479.6 6.898 0.10 48 42 1 1
648.3258 0.6 0.9 16.5 C32 H42 N9 O6 477.5 4.775 0.84 32 42 9 6
648.3271 -0.7 -1.1 21.5 C33 H38 N13 O2 477.3 4.635 0.97 33 38 13 2
648.3277 -1.3 -2.0 3.5 C20 H46 N11 O13 479.9 7.150 0.08 20 46 11 13
648.3245 1.9 2.9 11.5 C31 H46 N5 O10 477.8 5.130 0.59 31 46 5 10
19072019_299_5373
LD-5-1C_18072019_008 169 (4.353) AM2 (Ar,30000.0,556.39,0.00,LS 1)
1: TOF MS ES+
2.74e+007
100
%
0
648.3264
649.3304
648.2590
650.3372
651.3411
67.8722
165.0775
226.0927
279.1186
309.1686
406.1415
421.2005
500.2442
514.2675
606.3282
648.1792
693.3370
732.8245
834.1589
893.1547
1034.1814
1173.1720
m/z
HN
O
3af
For Help, press F1
2:47 PM
7/18/2019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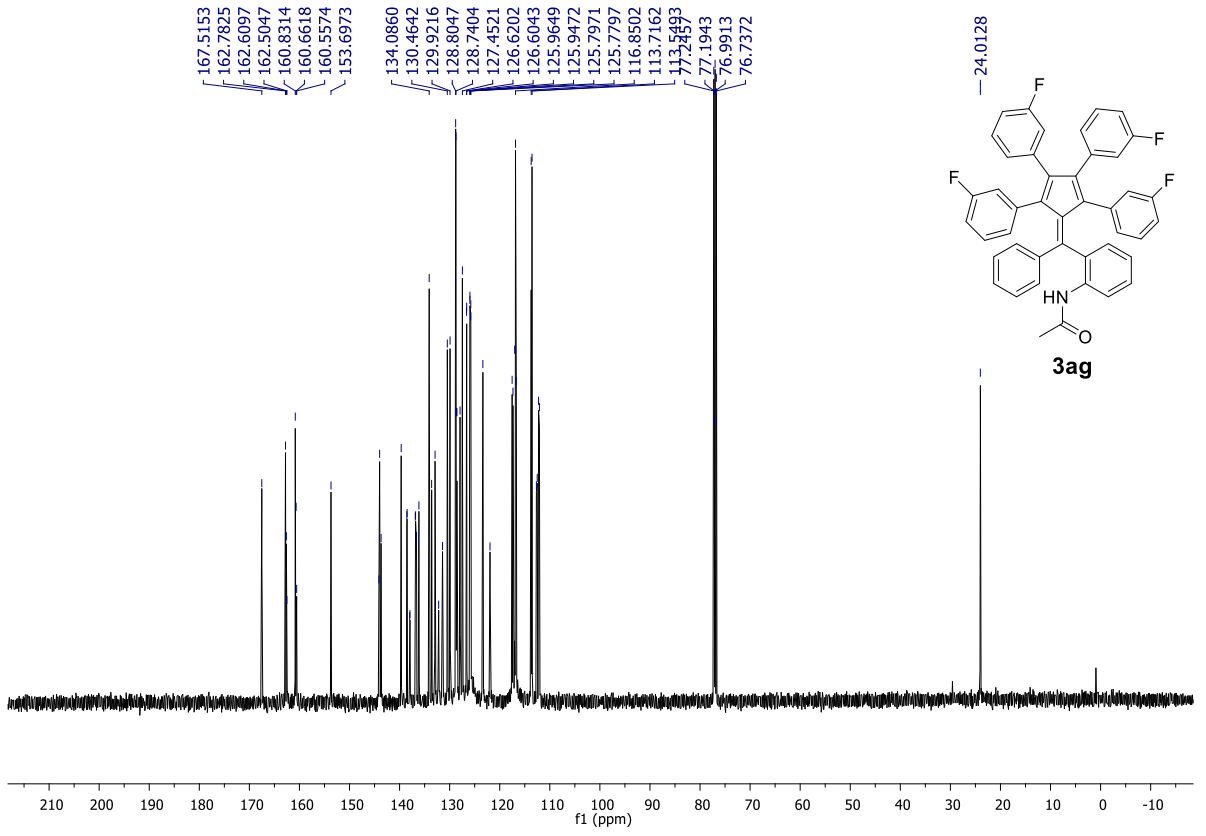
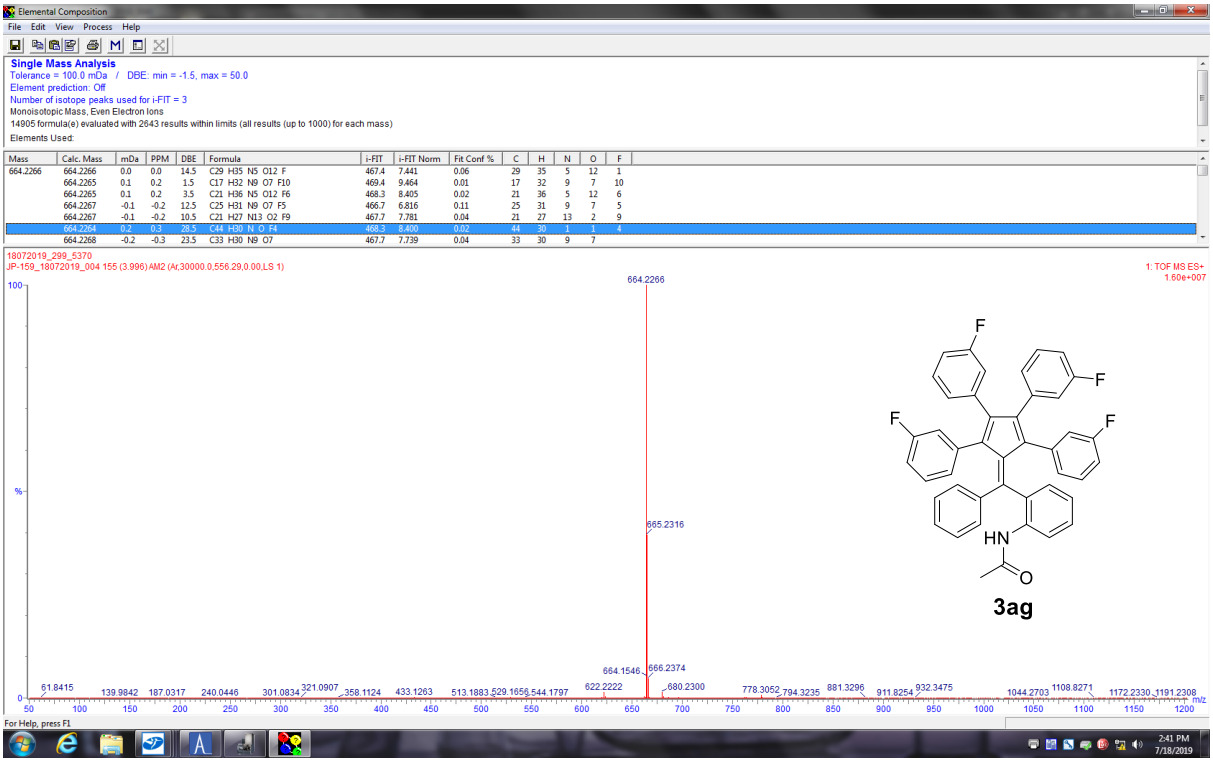
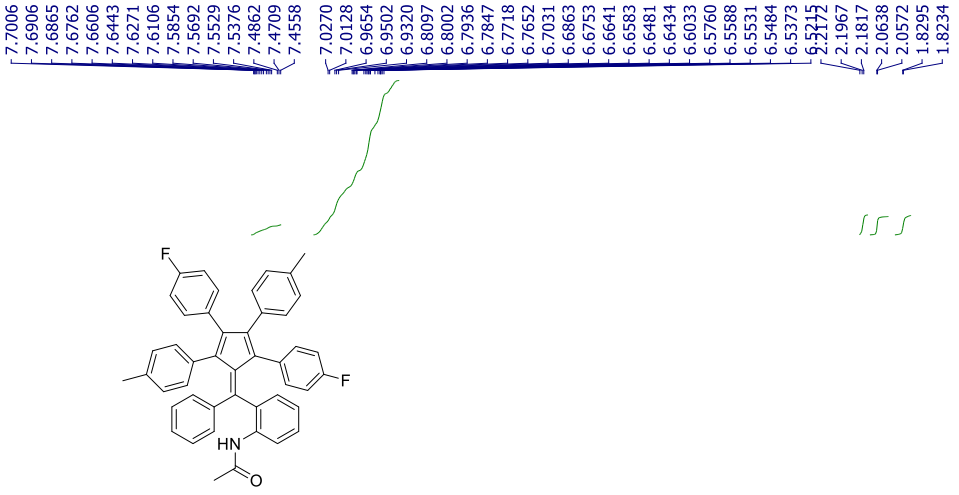
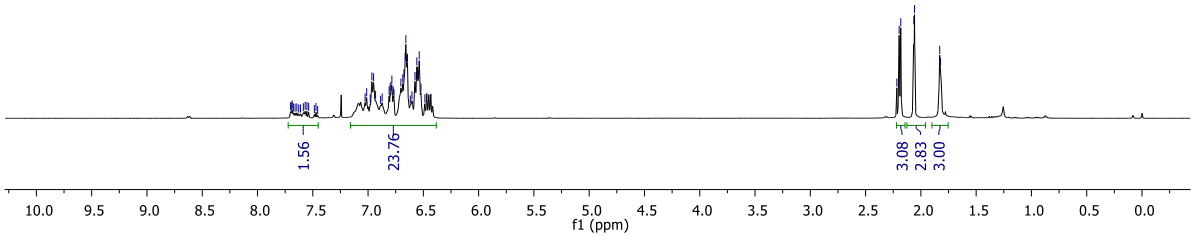
7.2601
7.2302
7.2161
7.0805
7.0659
7.0534
7.0379
7.0206
7.0031
6.9876
6.9765
6.9617
6.9495
6.9349
6.9116
6.8986
6.7884
6.7716
6.7531
6.7349
6.7195
6.5625
6.5364
6.5201
6.4946
6.4771
6.4604
6.4350
6.4182
6.4052
6.3865
1.7821
0.0048
F
F
F
F
HN
O
3ag
1.00
1.10
10.39
5.33
6.47
3.36
3.22
f1 (ppm)

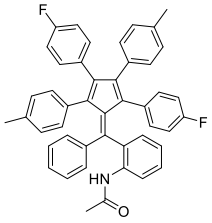

167.5153
162.7825
162.6097
162.5047
160.8314
160.6618
160.5574
153.6973
134.0860
130.4642
129.9216
128.8047
128.7404
127.4521
126.6202
126.6043
125.9649
125.9472
125.7971
125.7797
116.8502
113.7162
113.5493
77.2457
77.1943
76.9913
76.7372
24.0128
F
F
F
F
HN
O
3ag
f1 (ppm)





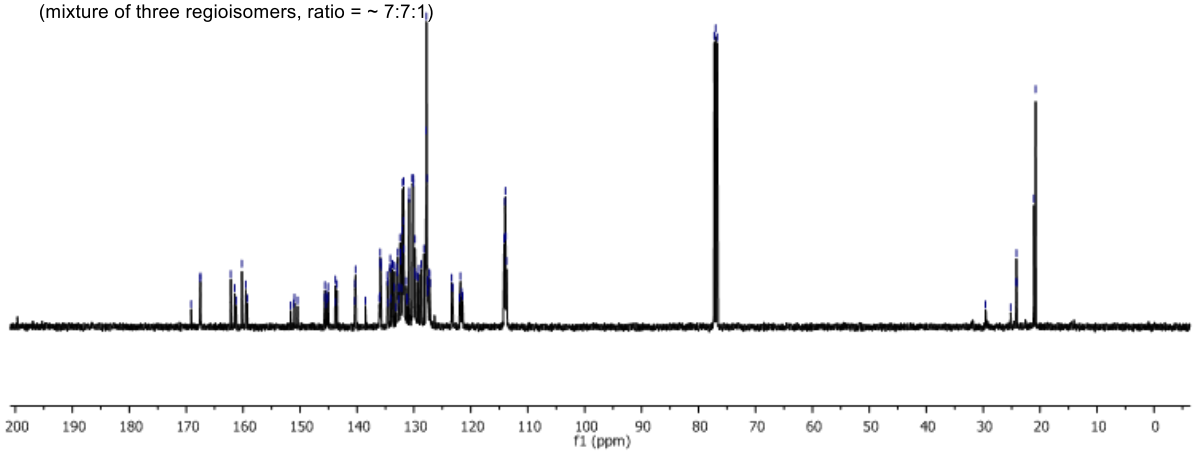
(mixture of three regioisomers, ratio = ~ 7:7:1)

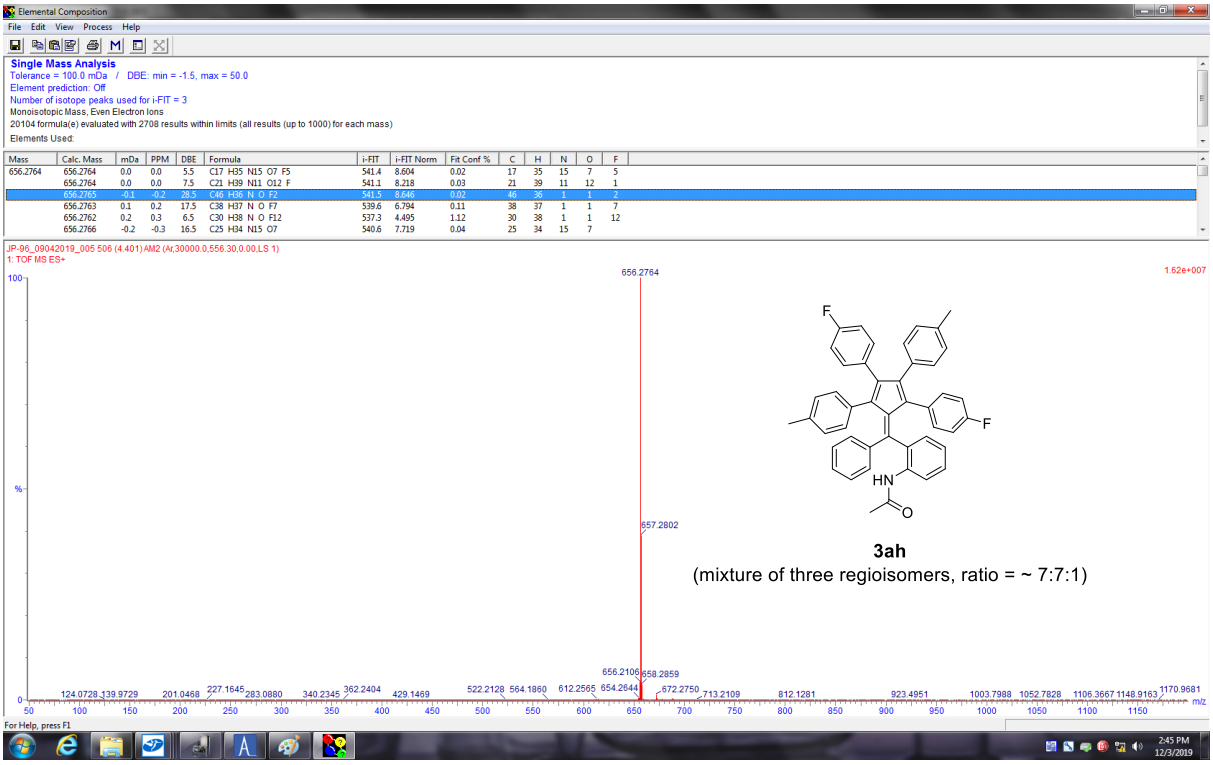




3ah

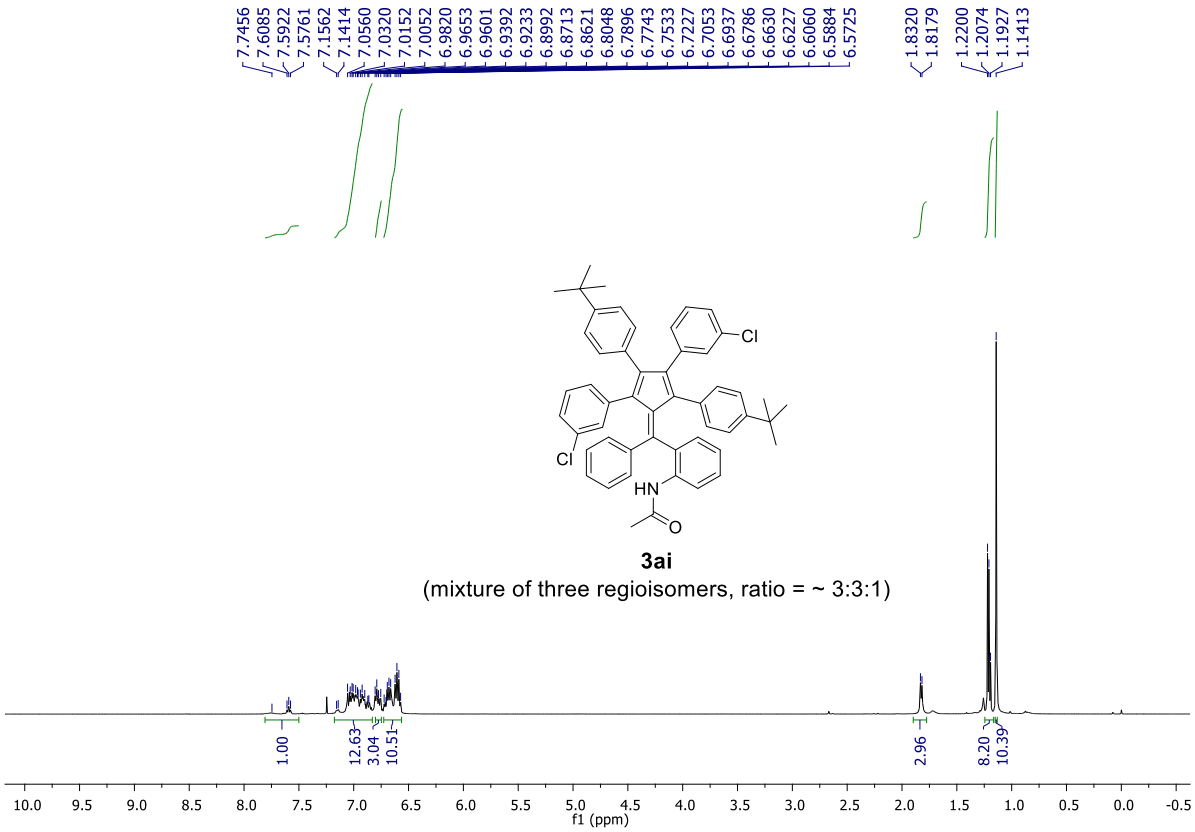
(mixture of three regioisomers, ratio = ~ 7:7:1)





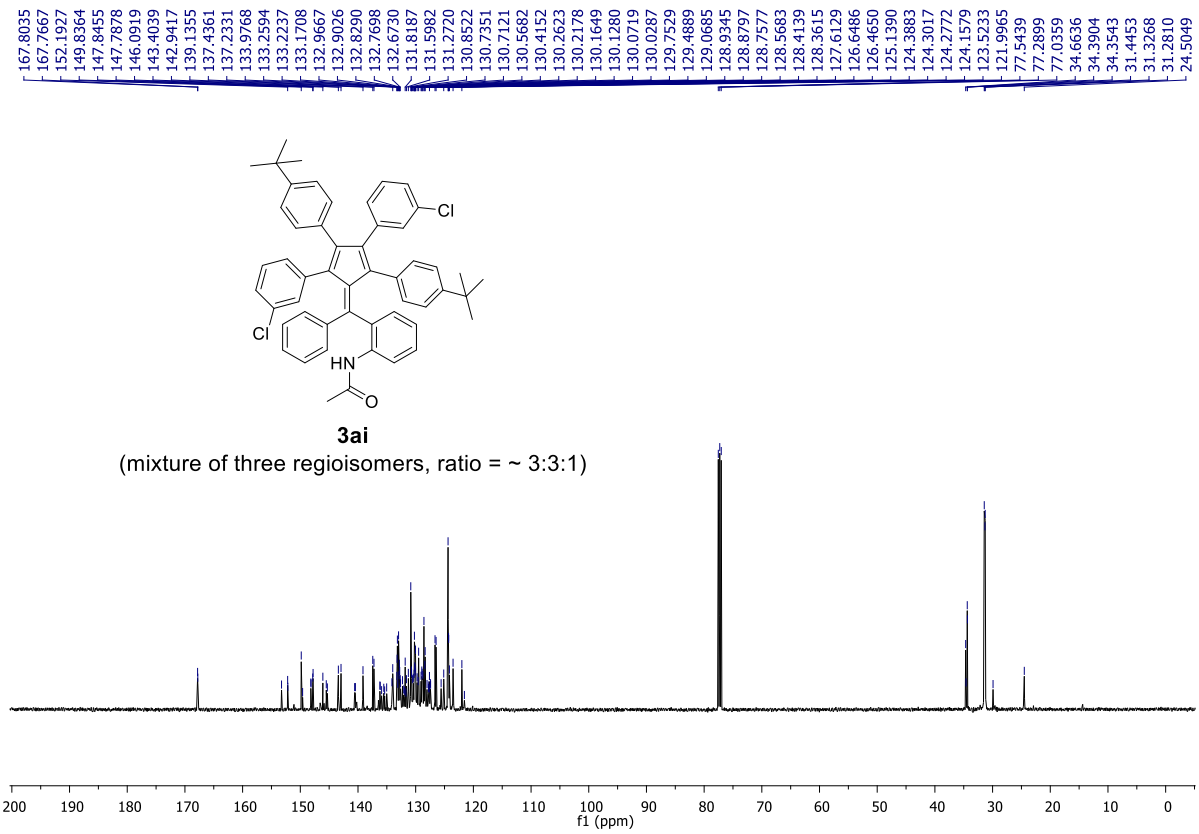
3ah

(mixture of three regioisomers, ratio = ~ 7:7:1)



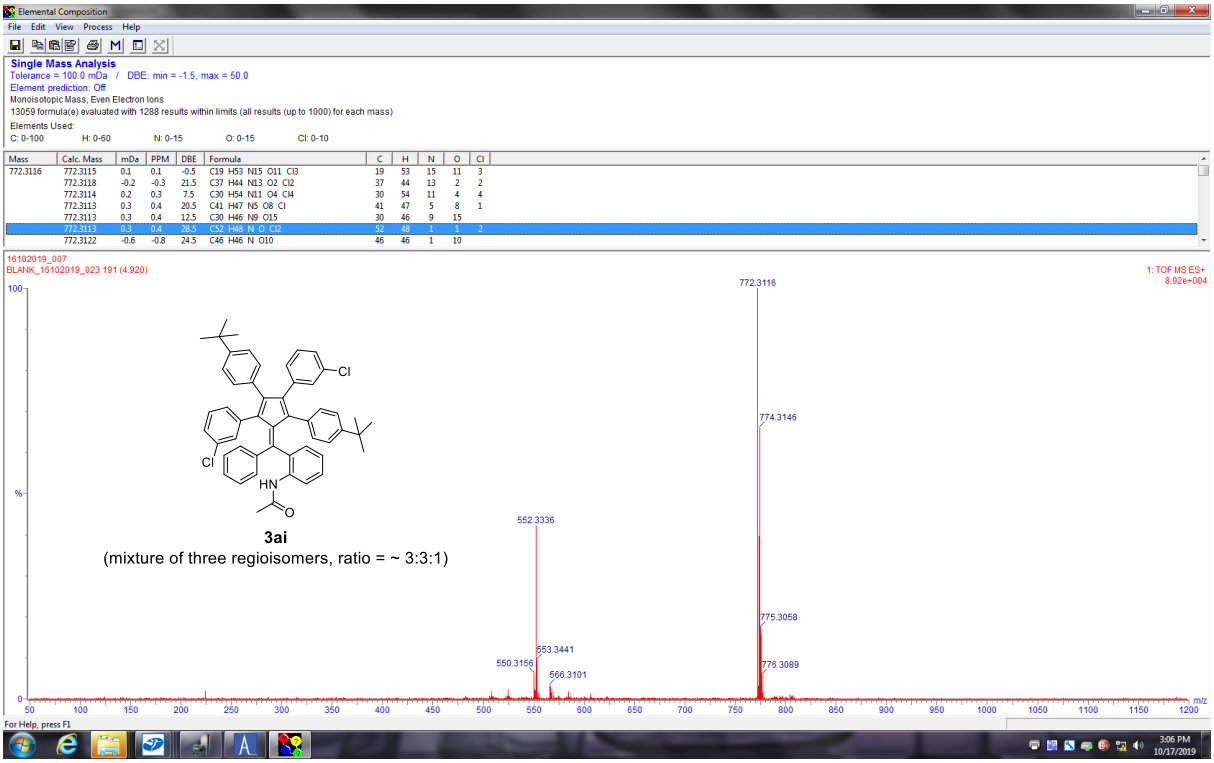
3ai

(mixture of three regioisomers, ratio = ~ 3:3:1)



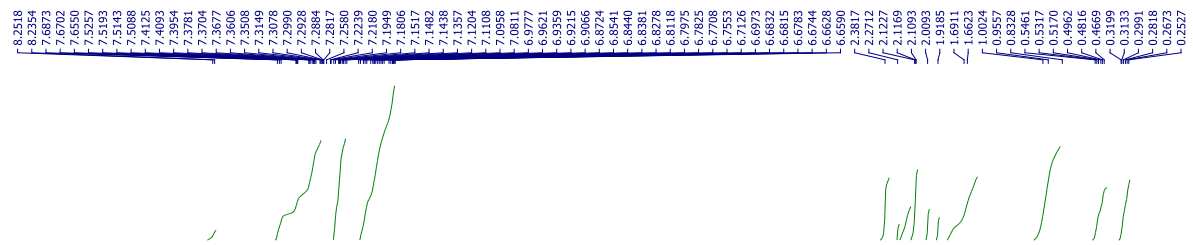
3ai

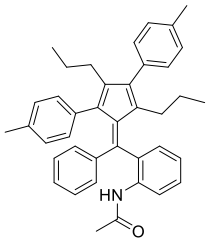
(mixture of three regioisomers, ratio = ~ 3:3:1)



3ai

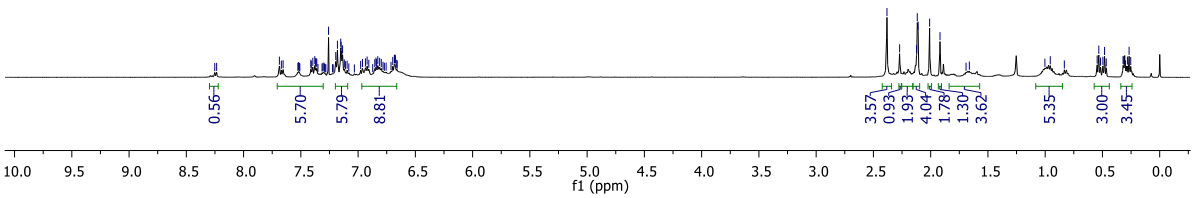
(mixture of three regioisomers, ratio = ~ 3:3:1)

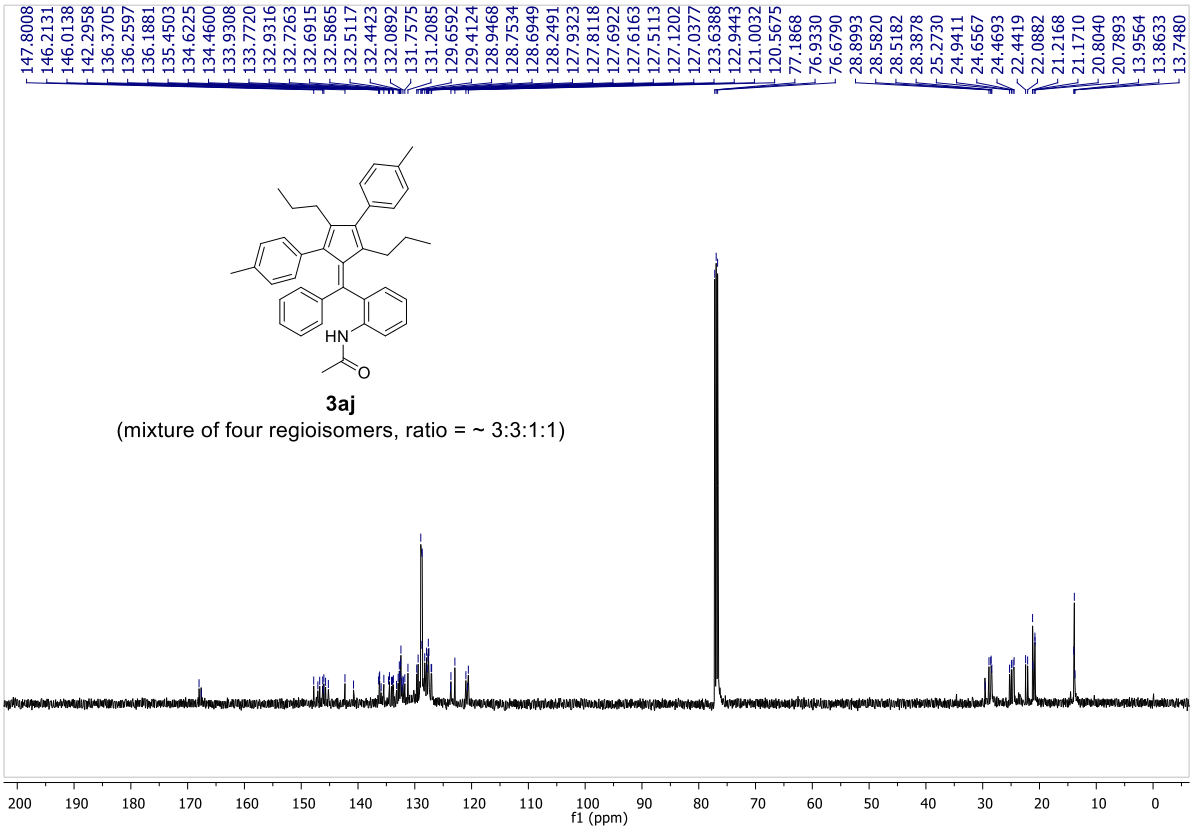




3aj

(mixture of four regioisomers, ratio = ~ 3:3:1:1)

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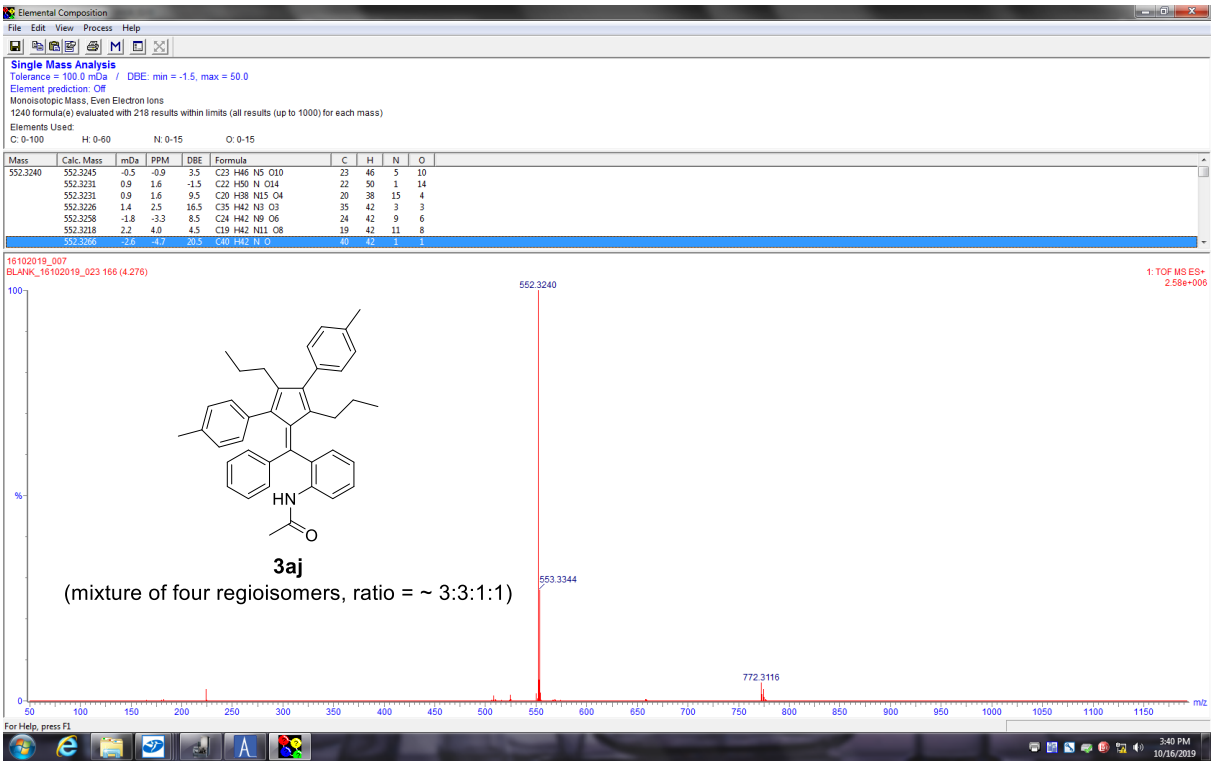
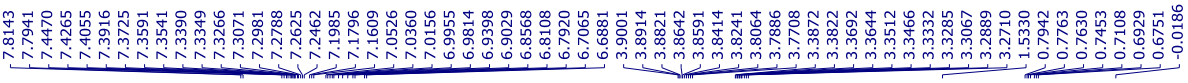
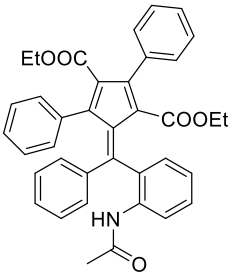
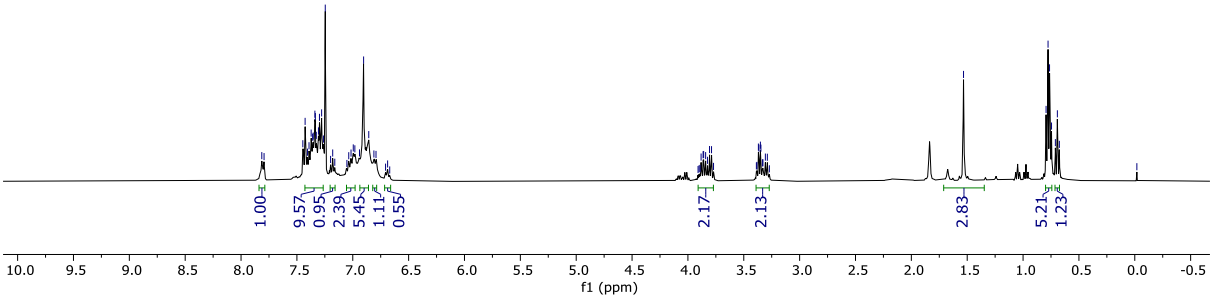
146.2131
146.0138
142.2958
136.3705
136.2597
136.1881
135.4503
134.6225
134.4600
133.9308
133.7720
132.9316
132.7263
132.6915
132.5865
132.5117
132.4423
132.0892
131.7575
131.2085
129.6592
129.4124
128.9468
128.7534
128.6949
128.2491
127.9323
127.8118
127.6922
127.6163
127.5113
127.1202
127.0377
123.6388
122.9443
121.0032
120.5675
77.1868
76.9330
76.6790
28.8993
28.5820
28.5182
28.3878
25.2730
24.9411
24.6567
24.4693
22.4419
22.0882
21.2168
21.1710
20.8040
20.7893
13.9564
13.8633
13.7480
HN
O
3aj
(mixture of four regioisomers, ratio = ~ 3:3:1:1)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

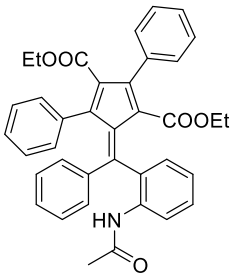

Elemental Composition
File Edit View Process Help
Single Mass Analysis
Tolerance = 100.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Monoisotopic Mass, Even Electron Ions
1240 formula(e) evaluated with 218 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-100 H: 0-60 N: 0-15 O: 0-15
Mass Calc. Mass mDa PPM DBE Formula C H N O
552.3240 552.3245 -0.5 -0.9 3.5 C23 H46 N5 O10 23 46 5 10
552.3231 0.9 1.6 -1.5 C22 H50 N O14 22 50 1 14
552.3231 0.9 1.6 9.5 C20 H38 N15 O4 20 38 15 4
552.3226 1.4 2.5 16.5 C35 H42 N3 O3 35 42 3 3
552.3258 -1.8 -3.3 8.5 C24 H42 N9 O6 24 42 9 6
552.3218 2.2 4.0 4.5 C19 H42 N11 O8 19 42 11 8
552.3266 -2.6 -4.7 20.5 C40 H42 N O 40 42 1 1
16102019_007
BLANK_16102019_023 166 (4.276)
1: TOF MS ES+
2.58e+006
552.3240
553.3344
772.3116
HN
O
3aj
(mixture of four regioisomers, ratio = ~ 3:3:1:1)
m/z
For Help, press F1
3:40 PM
10/16/2019



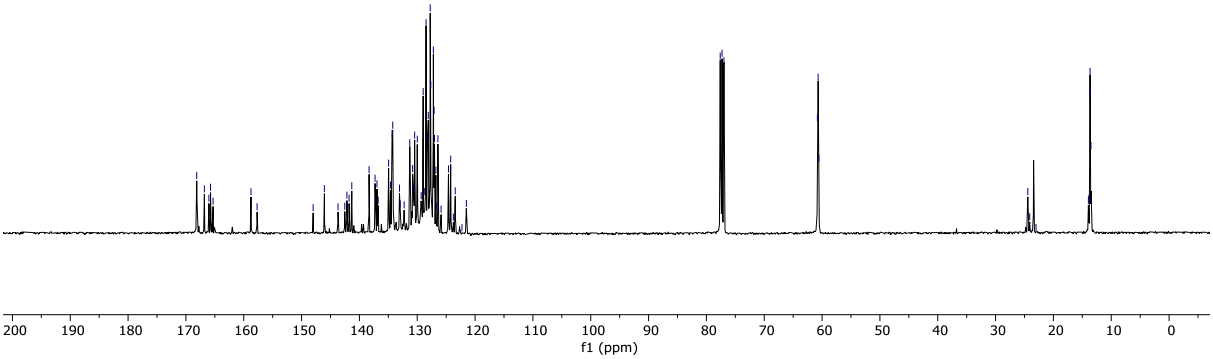


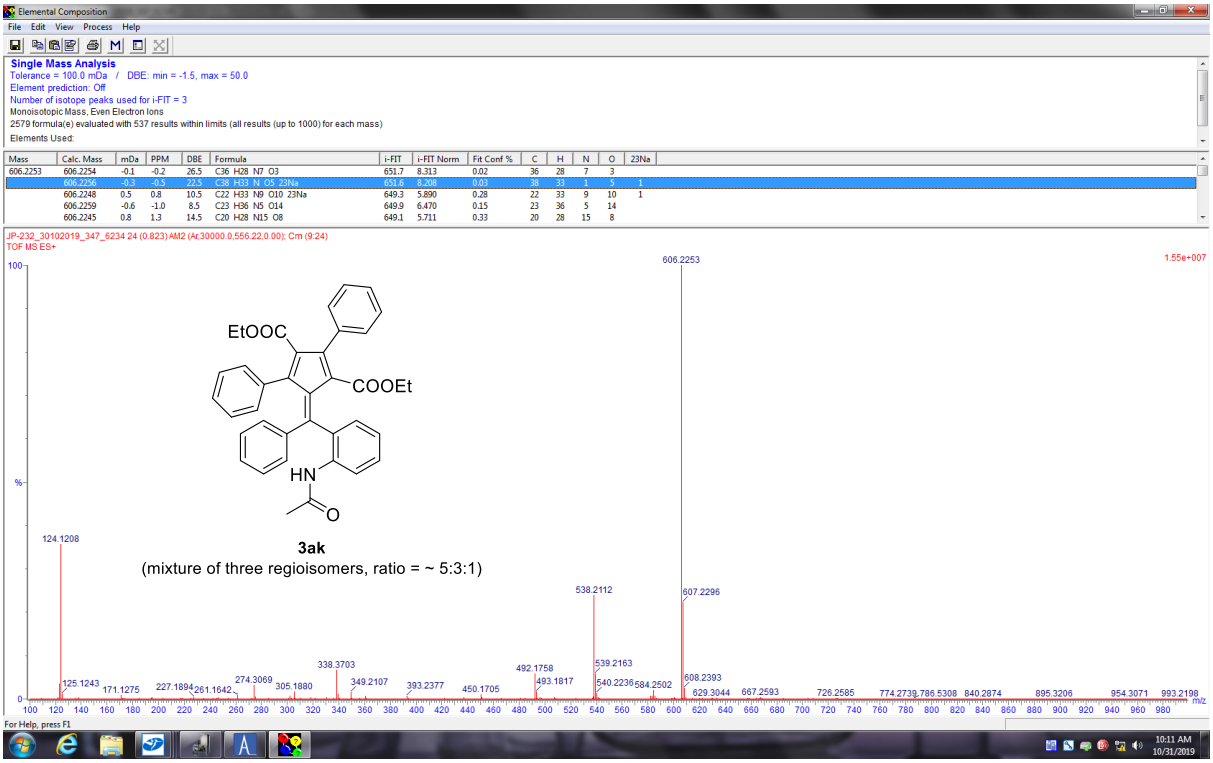
3ak
(mixture of three regioisomers, ratio = ~ 5:3:1)

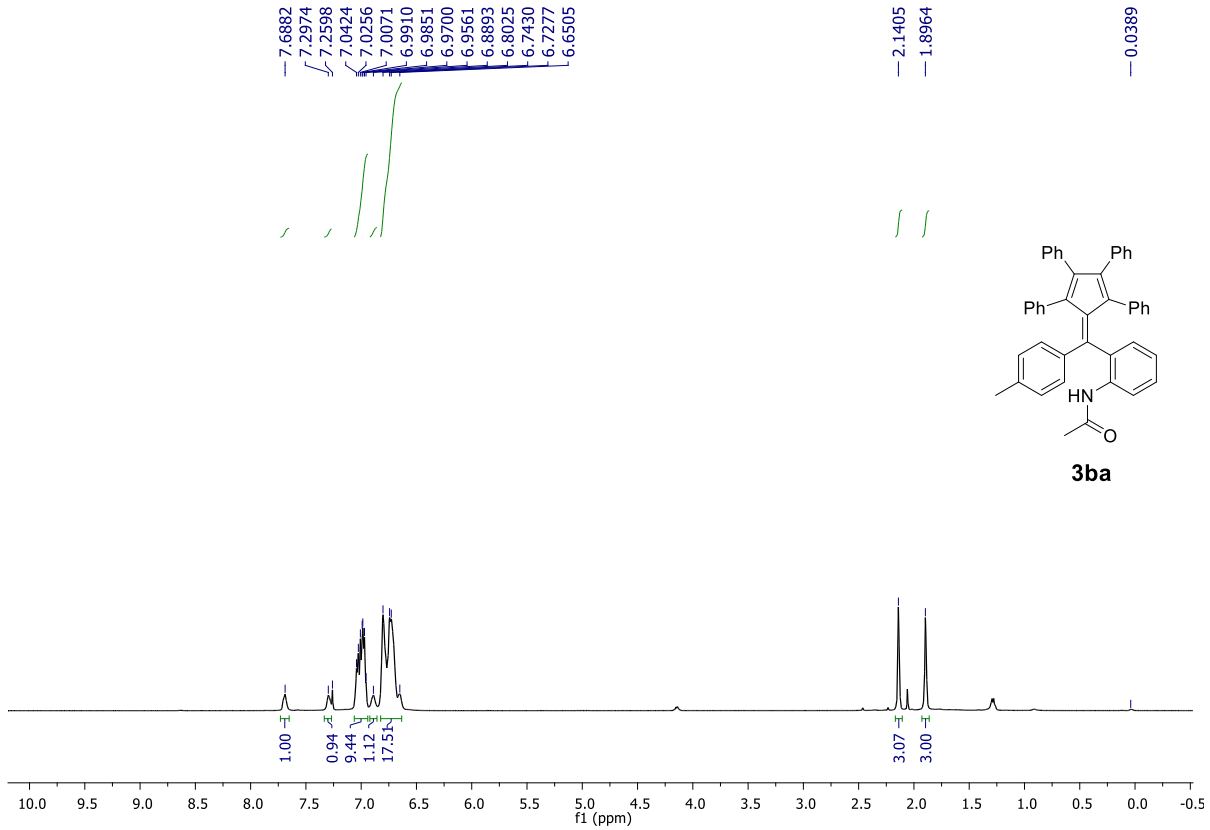


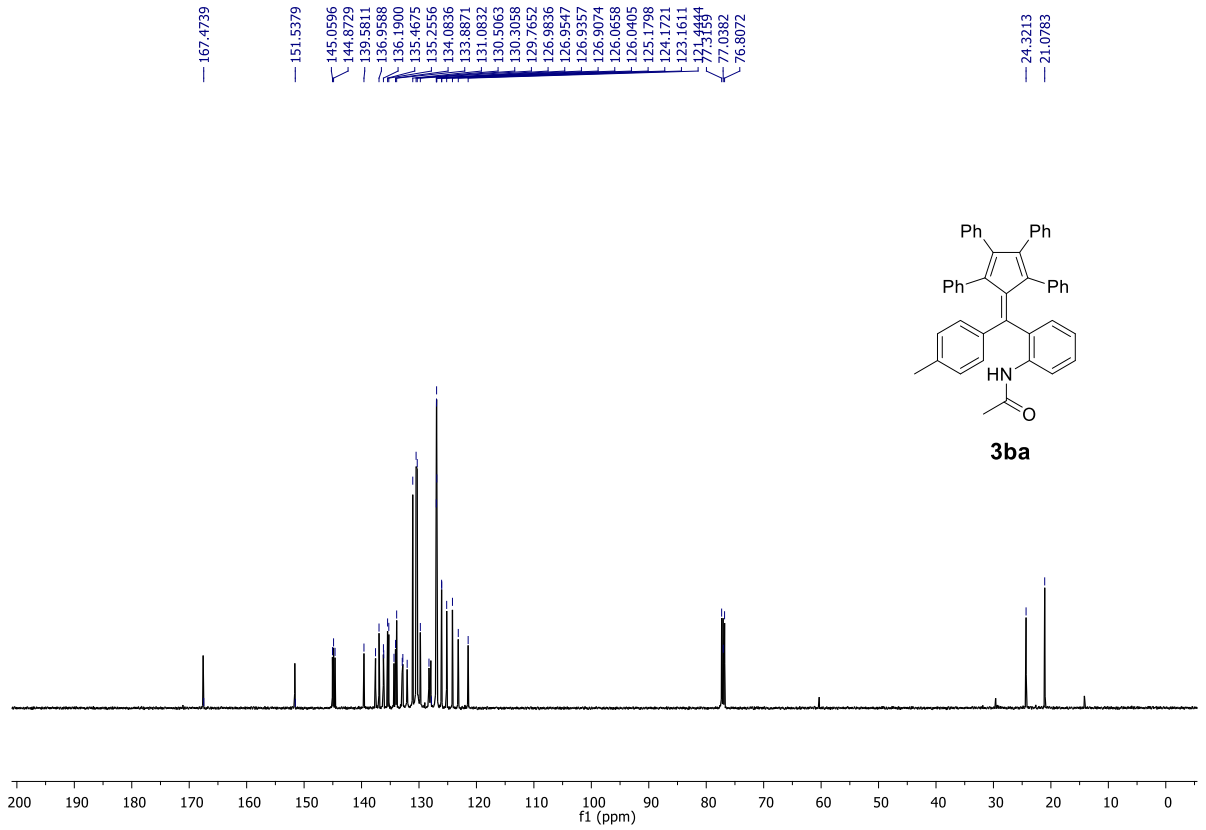


3ak
(mixture of three regioisomers, ratio = ~ 5:3:1)




Elemental Composition
Single Mass Analysis
Tolerance = 100.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3
Monoisotopic Mass, Even Electron Ions
2579 formula(e) evaluated with 537 results within limits (all results (up to 1000) for each mass)
Elements Used:
Mass Calc. Mass mDa PPM DBE Formula i-FIT i-FIT Norm Fit Conf % C H N O 23Na
606.2253 606.2254 -0.1 -0.2 26.5 C36 H28 N7 O3 651.7 8.313 0.02 36 28 7 3
606.2256 -0.3 -0.5 22.5 C38 H33 N O5 23Na 651.6 8.208 0.03 38 33 1 5 1
606.2248 0.5 0.8 10.5 C22 H33 N9 O10 23Na 649.3 5.890 0.28 22 33 9 10 1
606.2259 -0.6 -1.0 8.5 C23 H36 N5 O14 649.9 6.470 0.15 23 36 5 14
606.2245 0.8 1.3 14.5 C20 H28 N15 O8 649.1 5.711 0.33 20 28 15 8
JP-232_30102019_347_6234 24 (0.823) AM2 (Ar,30000.0,556.22,0.00); Cm (9:24)
TOF MS ES+
606.2253
1.55e+007
EtOOC
COOEt
HN
O
3ak
(mixture of three regioisomers, ratio = ~ 5:3:1)
124.1208
538.2112
607.2296
539.2163
338.3703
492.1758
608.2393
125.1243
171.1275
227.1894
261.1642
274.3069
305.1880
349.2107
393.2377
450.1705
493.1817
540.2236
584.2502
629.3044
667.2593
726.2585
774.2739
786.5308
840.2874
895.3206
954.3071
993.2198
m/z
For Help, press F1
10:11 AM
10/31/2019


7.6882
7.2974
7.2598
7.0424
7.0256
7.0071
6.9910
6.9851
6.9700
6.9561
6.8893
6.8025
6.7430
6.7277
6.6505
2.1405
1.8964
0.0389
Ph
Ph
Ph
Ph
HN
O
3ba
1.00
0.94
9.44
1.12
17.51
3.07
3.00
f1 (ppm)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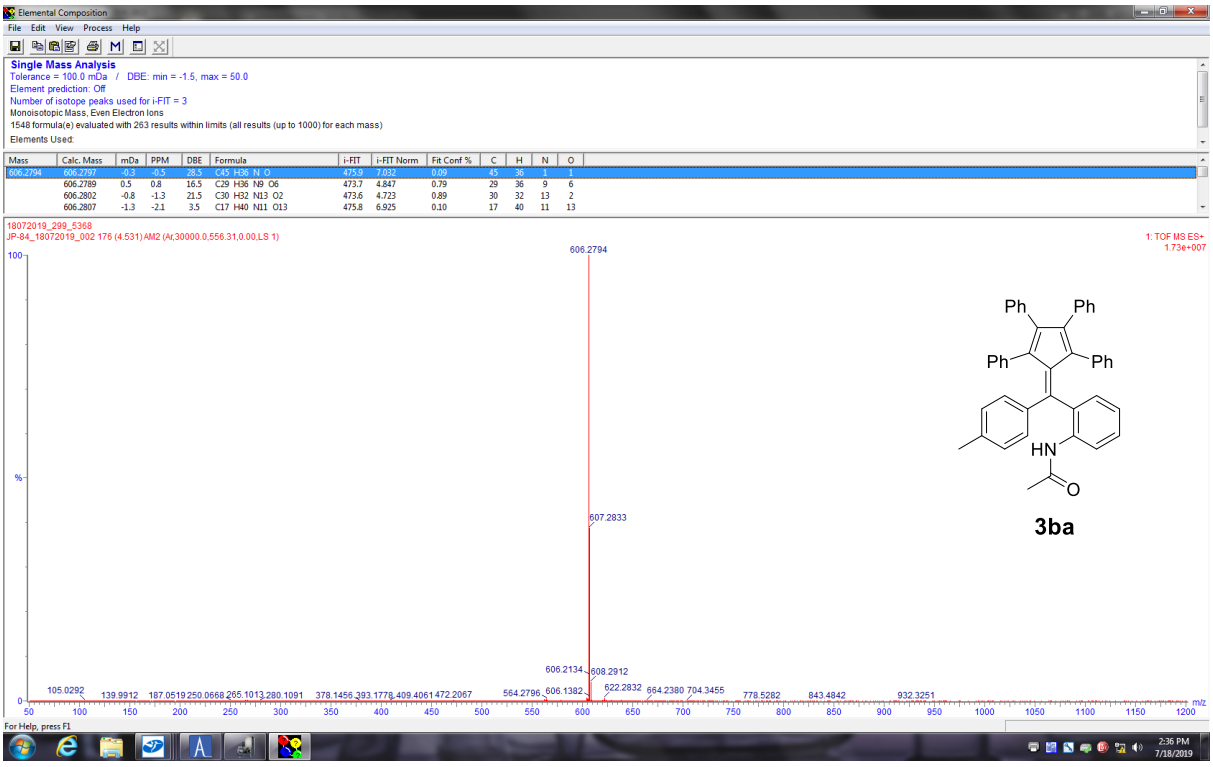
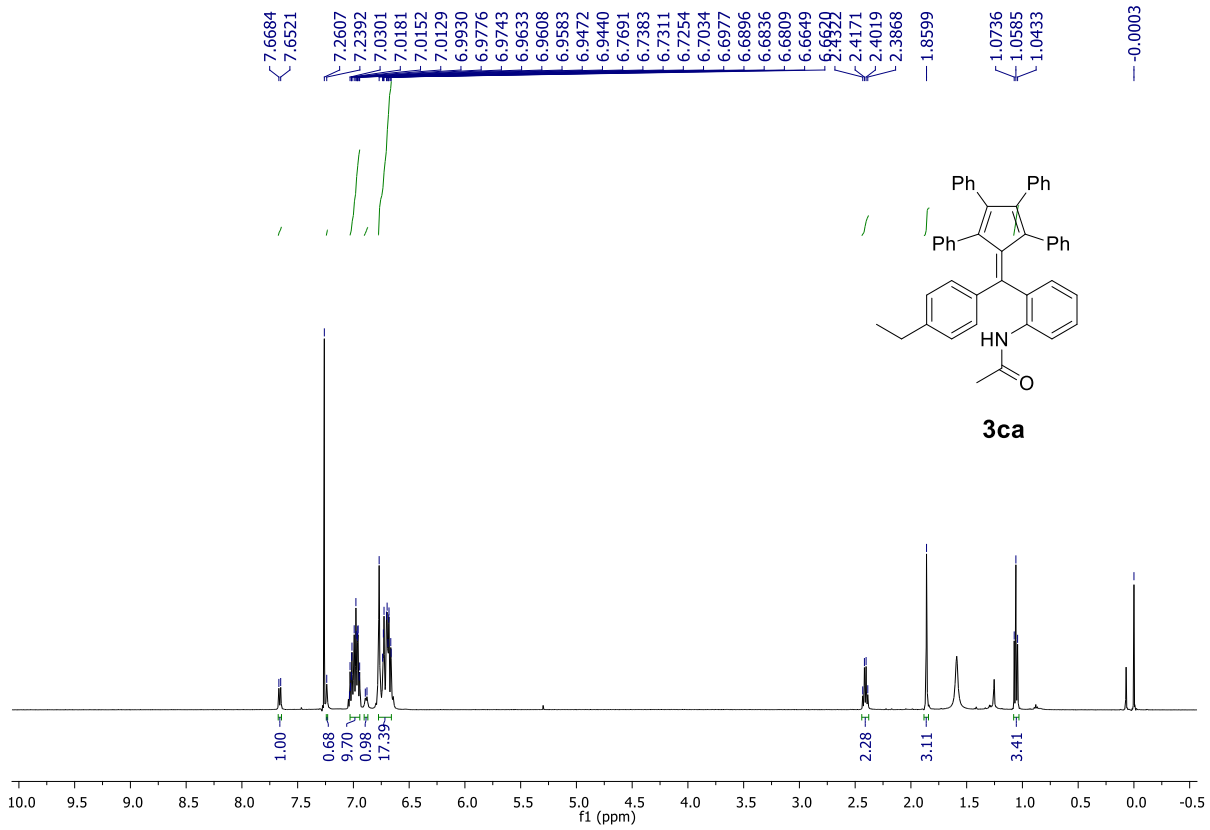
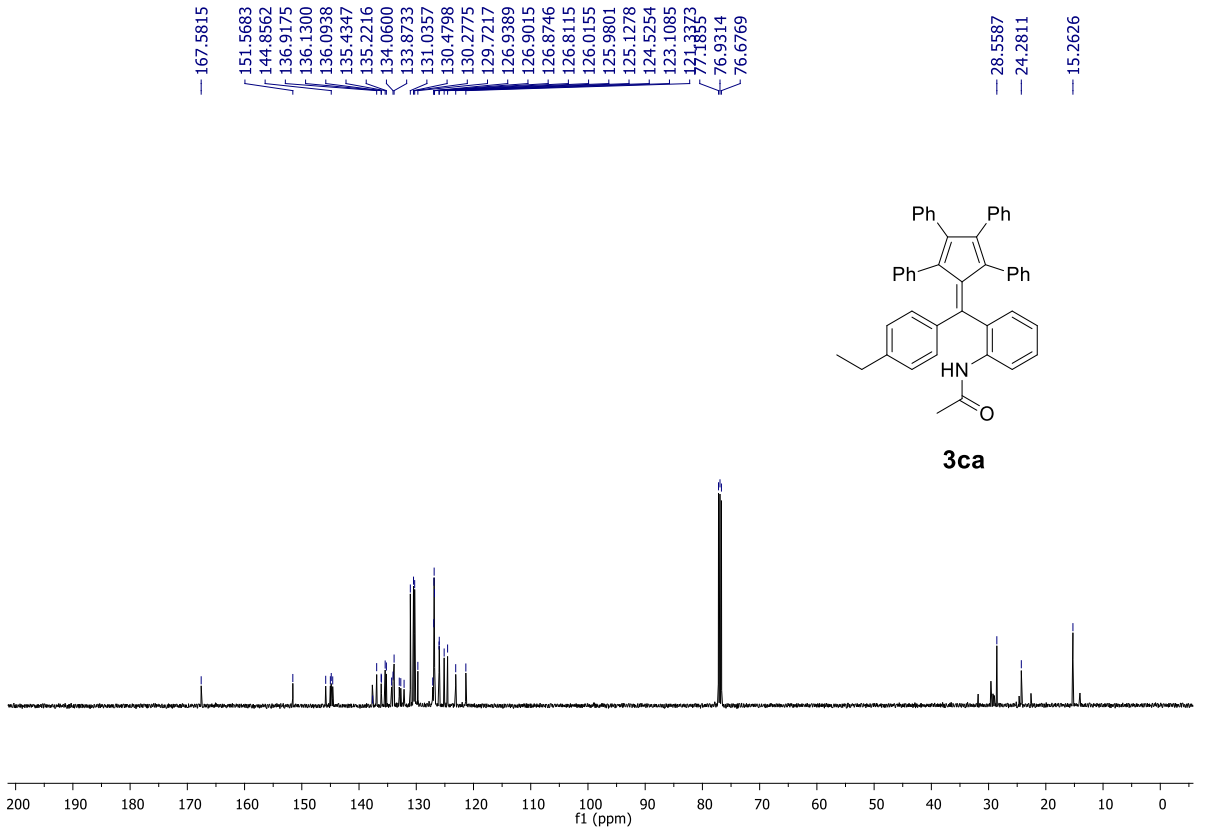
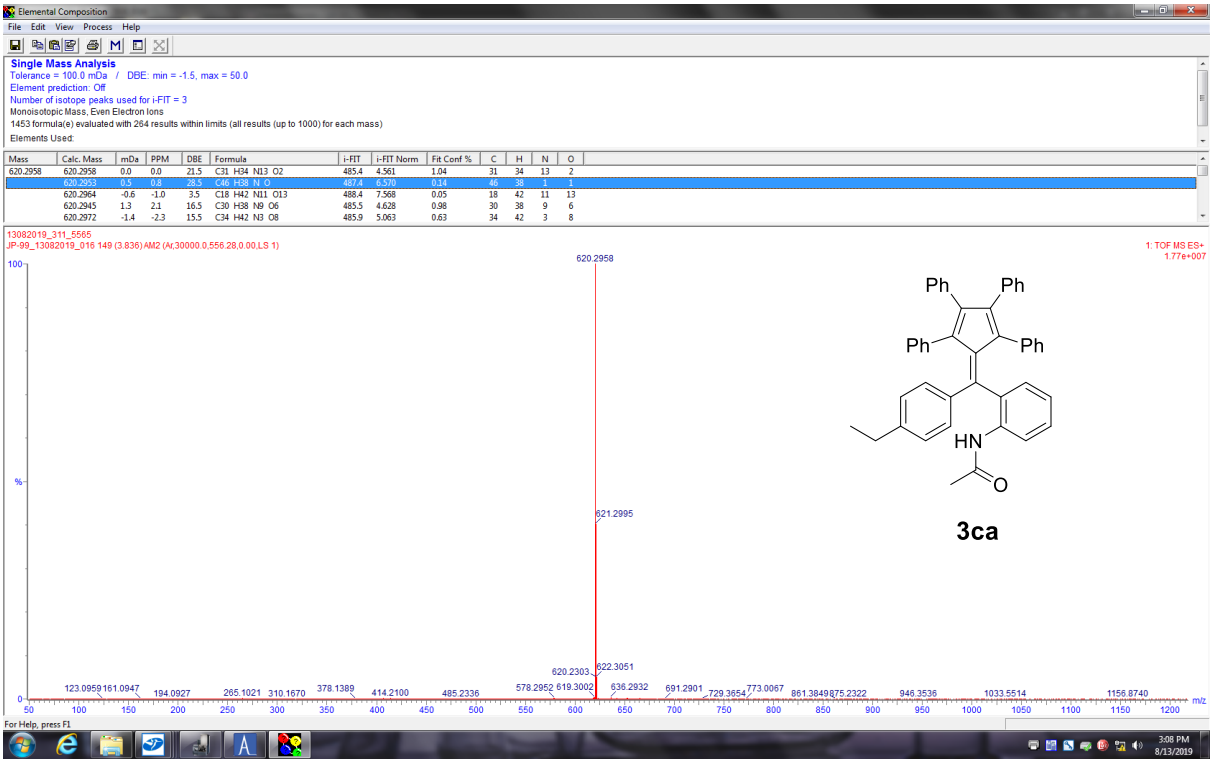
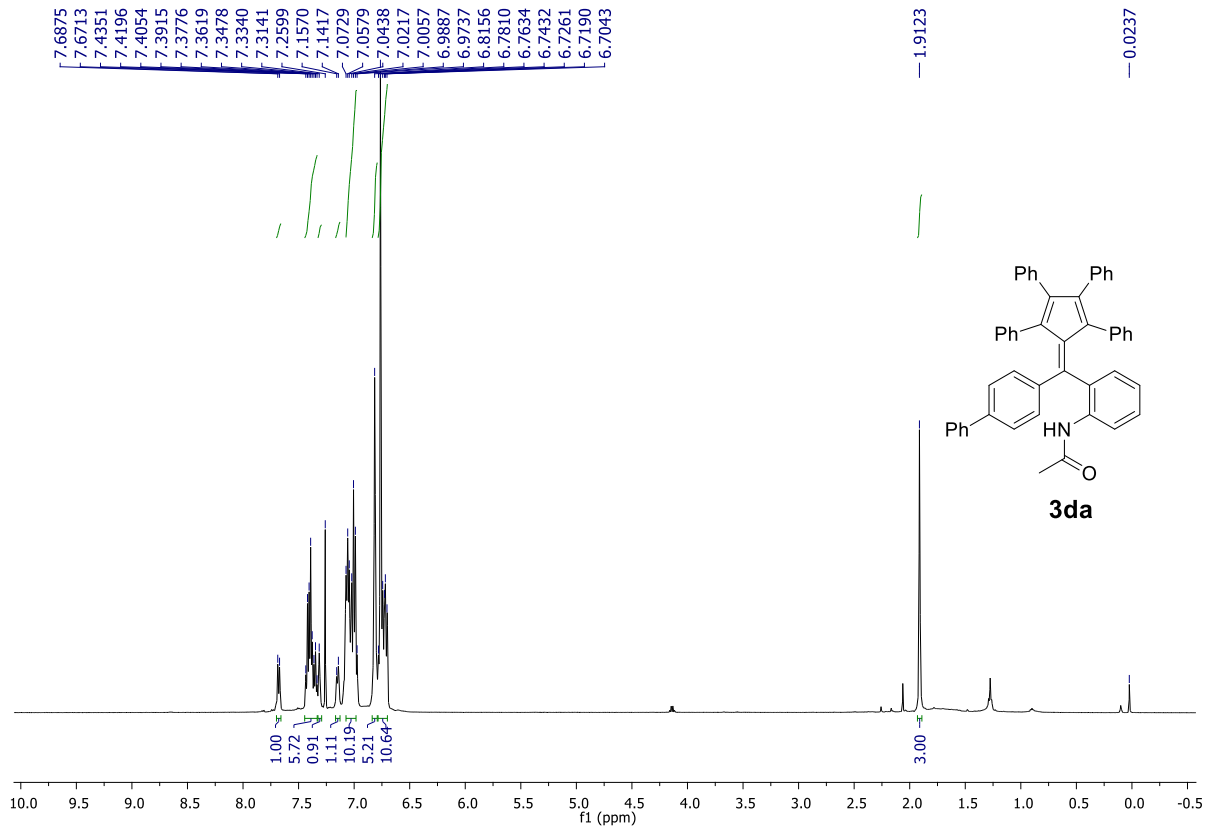
145.0596
144.8729
139.5811
136.9588
136.1900
135.4675
135.2556
134.0836
133.8871
131.0832
130.5063
130.3058
129.7652
126.9836
126.9547
126.9357
126.9074
126.0658
126.0405
125.1798
124.1721
123.1611
121.4444
77.3159
77.0382
76.8072
24.3213
21.0783
Ph
Ph
Ph
Ph
HN
O
3ba
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

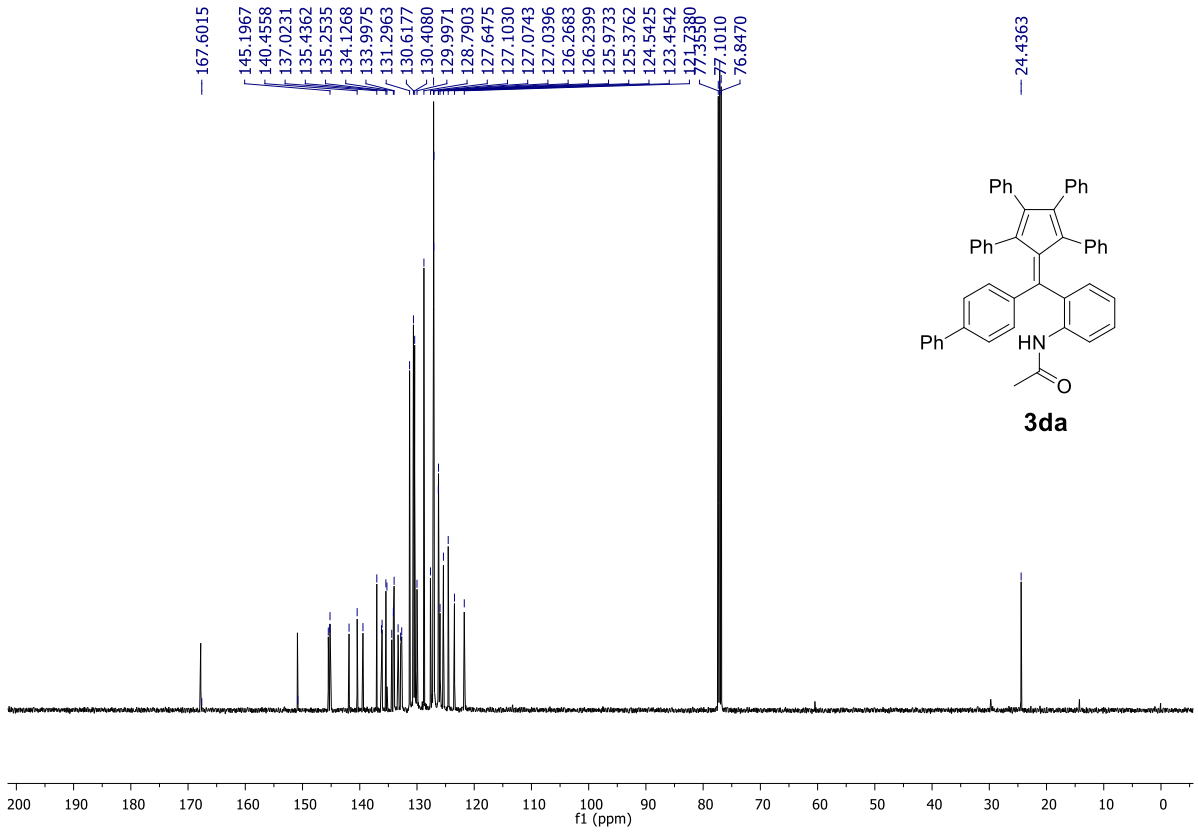

Elemental Composition
File Edit View Process Help
Single Mass Analysis
Tolerance = 100.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3
Monoisotopic Mass, Even Electron Ions
1548 formula(e) evaluated with 263 results within limits (all results (up to 1000) for each mass)
Elements Used:
Mass Calc. Mass mDa PPM DBE Formula i-FIT i-FIT Norm Fit Conf % C H N O
606.2794 606.2797 -0.3 -0.5 28.5 C45 H36 N O 475.9 7.032 0.09 45 36 1 1
606.2789 0.5 0.8 16.5 C29 H36 N9 O6 473.7 4.847 0.79 29 36 9 6
606.2802 -0.8 -1.3 21.5 C39 H32 N13 O2 473.6 4.723 0.89 30 32 13 2
606.2807 -1.3 -2.1 3.5 C17 H40 N11 O13 475.8 6.925 0.10 17 40 11 13
19072019_299_5368
JP-84_18072019_002 176 (4.531) AM2 (Ar,30000.0,556.31,0.00,LS 1)
1: TOF MS ES+
1.73e+007
606.2794
607.2833
606.2134
608.2912
622.2832
664.2380
704.3455
105.0292
139.9912
187.0519
250.0668
265.1013
280.1091
378.1456
393.1778
409.4061
472.2067
564.2796
606.1382
778.5282
843.4842
932.3251
m/z
For Help, press F1
Ph
Ph
Ph
Ph
HN
O
3ba
2:36 PM
7/18/2019

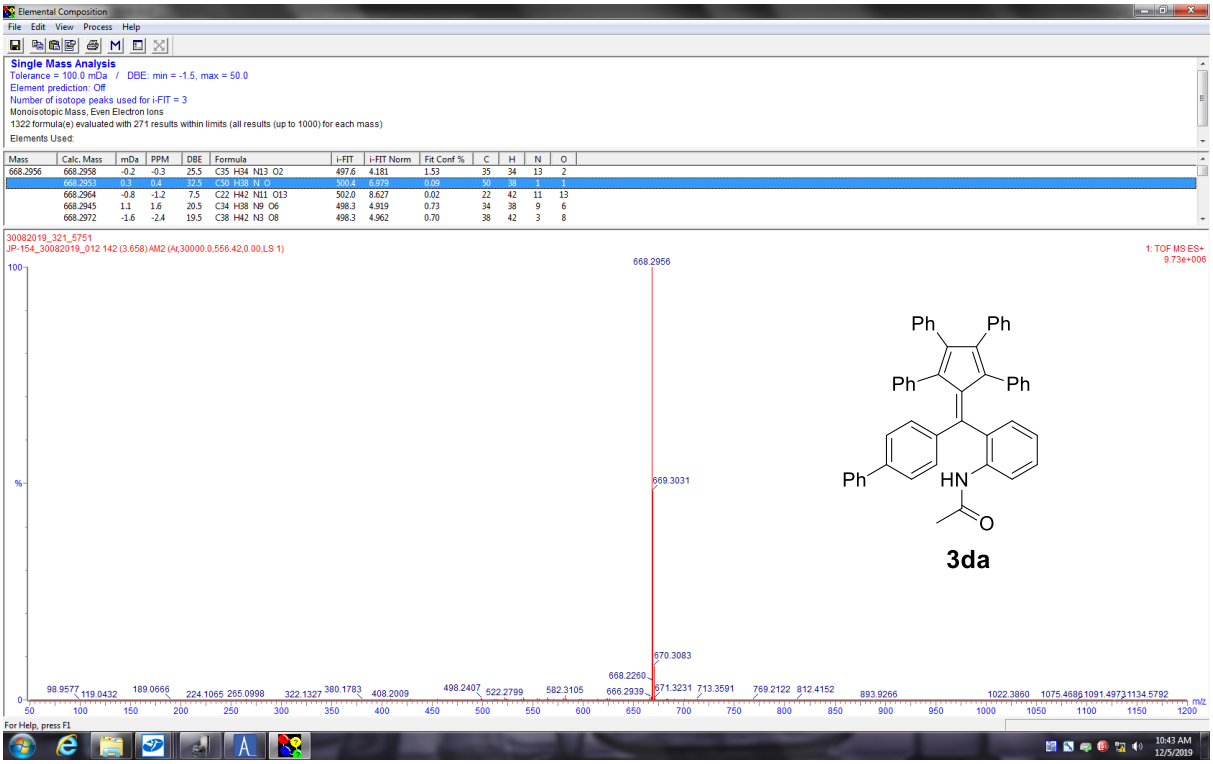

7.6684
7.6521
7.2607
7.2392
7.0301
7.0181
7.0152
7.0129
6.9930
6.9776
6.9743
6.9633
6.9608
6.9583
6.9472
6.9440
6.7691
6.7383
6.7311
6.7254
6.7034
6.6977
6.6896
6.6836
6.6809
6.6649
6.6620
2.4322
2.4171
2.4019
2.3868
1.8599
1.0736
1.0585
1.0433
-0.0003
Ph
Ph
Ph
Ph
HN
O
3ca
1.00
0.68
9.70
0.98
17.39
2.28
3.11
3.41
f1 (ppm)

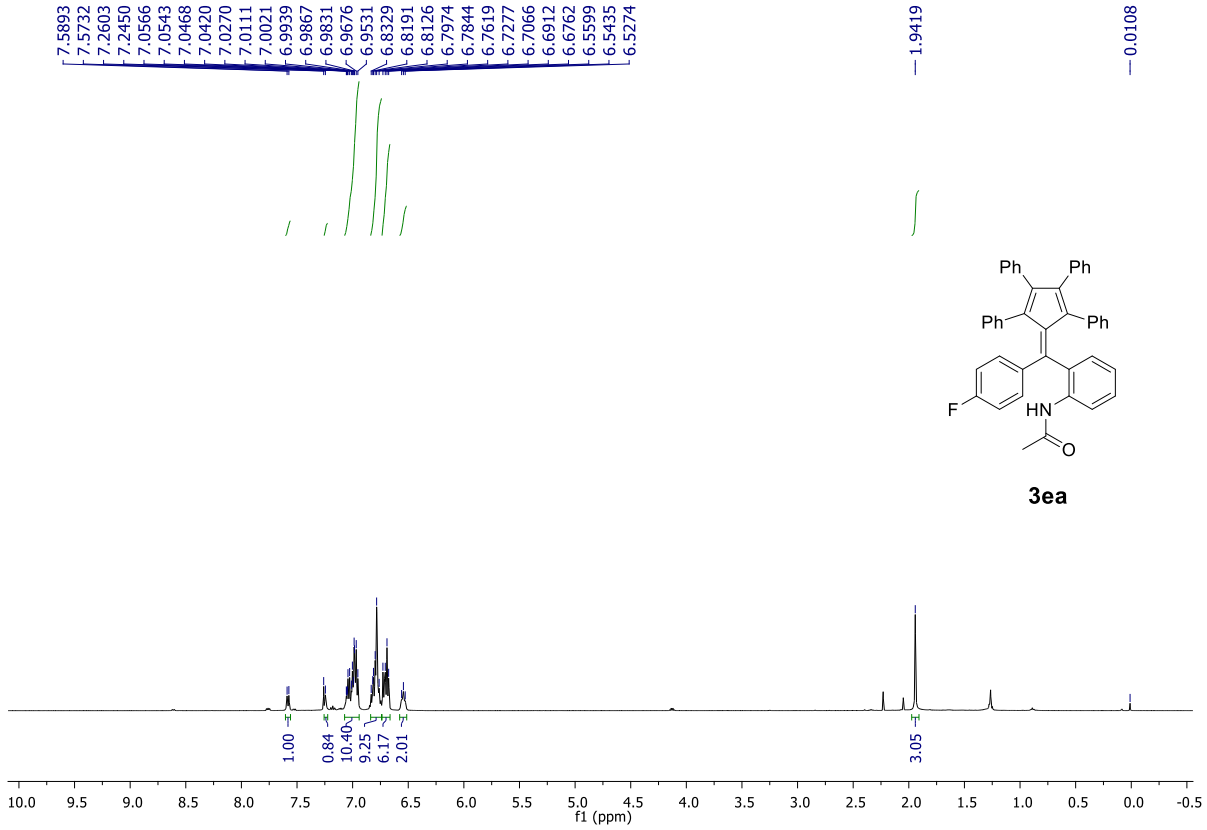

167.5815
151.5683
144.8562
136.9175
136.1300
136.0938
135.4347
135.2216
134.0600
133.8733
131.0357
130.4798
130.2775
129.7217
126.9389
126.9015
126.8746
126.8115
126.0155
125.9801
125.1278
124.5254
123.1085
77.1855
76.9314
76.6769
28.5587
24.2811
15.2626
Ph
Ph
Ph
Ph
HN
O
3ca
f1 (ppm)

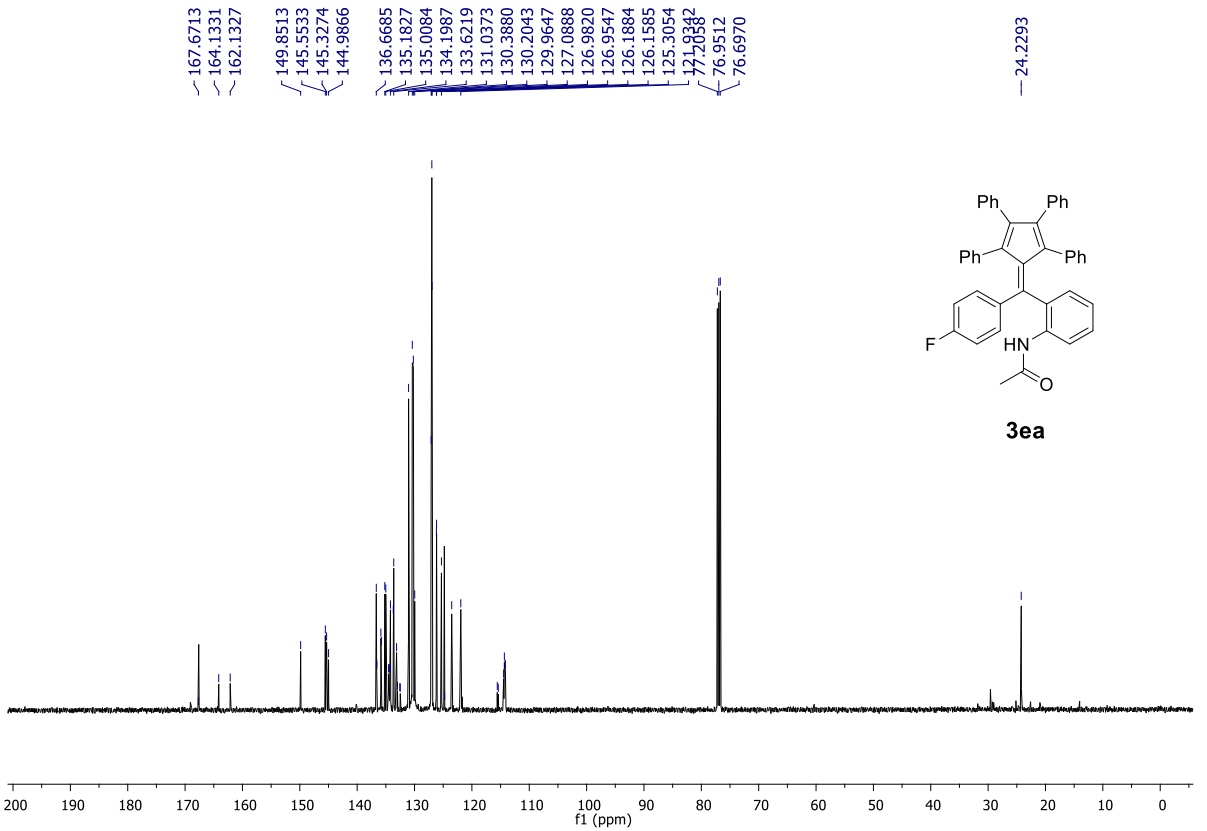

Elemental Composition
Single Mass Analysis
Tolerance = 100.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3
Monoisotopic Mass, Even Electron Ions
1453 formula(e) evaluated with 264 results within limits (all results (up to 1000) for each mass)
Elements Used:
620.2958
621.2995
622.3051
Ph
Ph
Ph
Ph
HN
O
3ca


7.6875
7.6713
7.4351
7.4196
7.4054
7.3915
7.3776
7.3619
7.3478
7.3340
7.3141
7.2599
7.1570
7.1417
7.0729
7.0579
7.0438
7.0217
7.0057
6.9887
6.9737
6.8156
6.7810
6.7634
6.7432
6.7261
6.7190
6.7043
1.9123
0.0237
1.00
5.72
0.91
1.11
10.19
5.21
10.64
3.00
f1 (ppm)
Ph
Ph
Ph
Ph
Ph
HN
O
3da


167.6015
145.1967
140.4558
137.0231
135.4362
135.2535
134.1268
133.9975
131.2963
130.6177
130.4080
129.9971
128.7903
127.6475
127.1030
127.0743
127.0396
126.2683
126.2399
125.9733
125.3762
124.5425
123.4542
77.3550
77.1010
76.8470
24.4363
Ph
Ph
Ph
Ph
Ph
HN
O
3da
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


Elemental Composition
File Edit View Process Help
Single Mass Analysis
Tolerance = 100.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3
Monoisotopic Mass, Even Electron Ions
1322 formula(e) evaluated with 271 results within limits (all results (up to 1000) for each mass)
Elements Used:
Mass | Calc. Mass | mDa | PPM | DBE | Formula | i-FIT | i-FIT Norm | Fit Conf % | C | H | N | O
668.2956 | 668.2958 | -0.2 | -0.3 | 25.5 | C35 H34 N13 O2 | 497.6 | 4.181 | 1.53 | 35 | 34 | 13 | 2
668.2953 | 0.3 | 0.4 | 32.5 | C50 H38 N O | 500.4 | 6.979 | 0.09 | 50 | 38 | 1 | 1
668.2964 | -0.8 | -1.2 | 7.5 | C22 H42 N11 O13 | 502.0 | 8.627 | 0.02 | 22 | 42 | 11 | 13
668.2945 | 1.1 | 1.6 | 20.5 | C34 H38 N9 O6 | 498.3 | 4.919 | 0.73 | 34 | 38 | 9 | 6
668.2972 | -1.6 | -2.4 | 19.5 | C38 H42 N3 O8 | 498.3 | 4.962 | 0.70 | 38 | 42 | 3 | 8
30082019_321_5751
JP-154_30082019_012 142 (3.658) AM2 (Ar,30000.0,556.42,0.00,LS 1)
1: TOF MS ES+
9.73e+006
100
%
0
668.2956
669.3031
670.3083
668.2260
666.2939
671.3231
713.3591
769.2122
812.4152
893.9266
1022.3860
1075.4686
1091.4973
1134.5792
98.9577
119.0432
189.0666
224.1065
265.0998
322.1327
380.1783
408.2009
498.2407
522.2799
582.3105
50
100
150
200
250
300
350
400
450
500
550
600
650
700
750
800
850
900
950
1000
1050
1100
1150
1200
m/z
Ph
Ph
Ph
Ph
Ph
HN
O
3da
For Help, press F1
10:43 AM
12/5/2019


7.5893
7.5732
7.2603
7.2450
7.0566
7.0543
7.0468
7.0420
7.0270
7.0111
7.0021
6.9939
6.9867
6.9831
6.9676
6.9531
6.8329
6.8191
6.8126
6.7974
6.7844
6.7619
6.7277
6.7066
6.6912
6.6762
6.5599
6.5435
6.5274
1.9419
0.0108
Ph
Ph
Ph
Ph
F
HN
O
3ea
1.00
0.84
10.40
9.25
6.17
2.01
3.05
f1 (ppm)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164.1331
162.1327
149.8513
145.5533
145.3274
144.9866
136.6685
135.1827
135.0084
134.1987
133.6219
131.0373
130.3880
130.2043
129.9647
127.0888
126.9820
126.9547
126.1884
126.1585
125.3054
77.2038
76.9512
76.6970
24.2293
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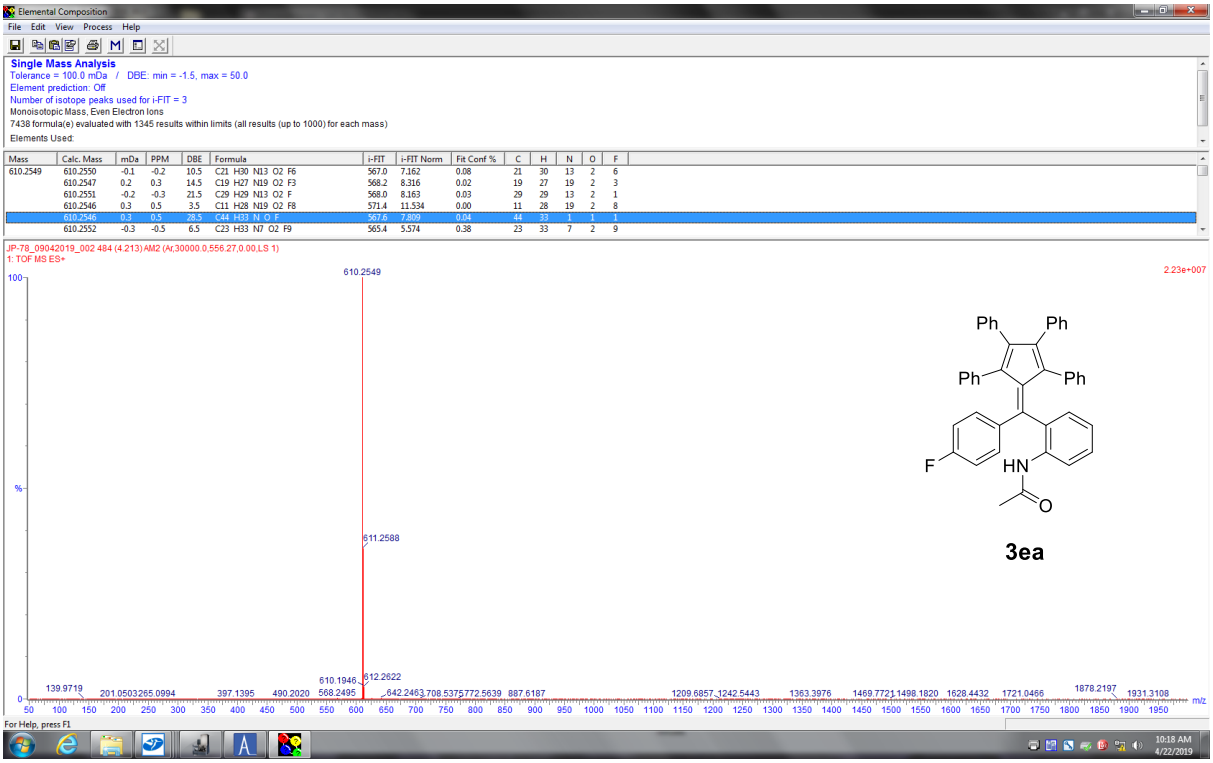
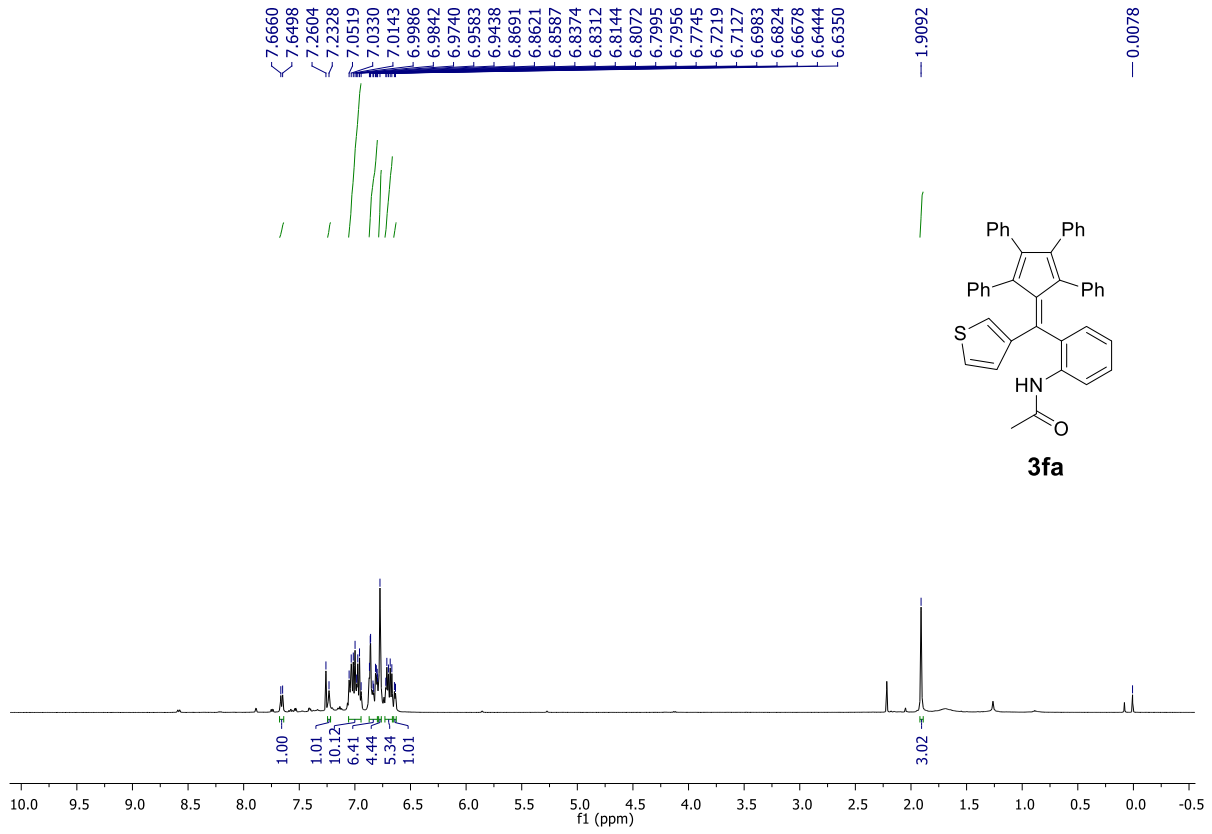
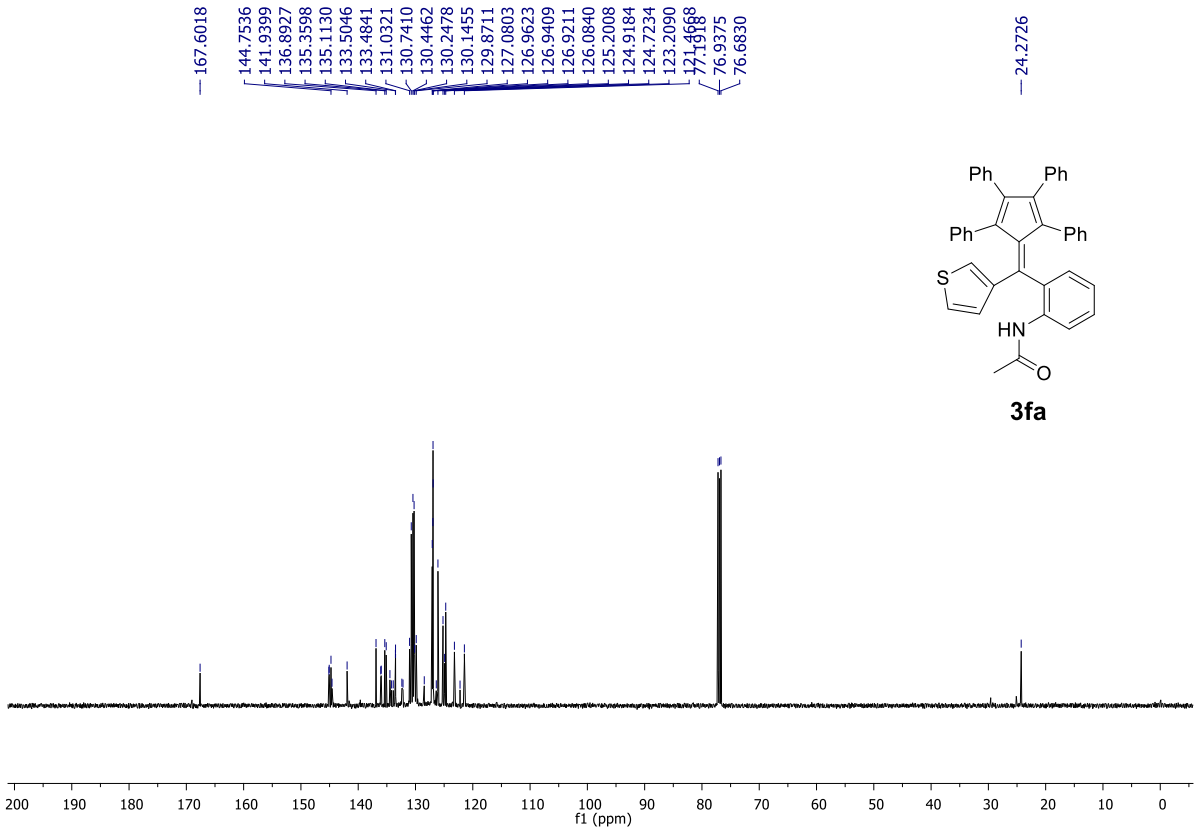
Ph
Ph
Ph
F
HN
O
3ea
f1 (ppm)


Elemental Composition
File Edit View Process Help
Single Mass Analysis
Tolerance = 100.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3
Monoisotopic Mass, Even Electron Ions
7438 formula(e) evaluated with 1345 results within limits (all results (up to 1000) for each mass)
Elements Used:
Mass Calc. Mass mDa PPM DBE Formula i-FIT i-FIT Norm Fit Conf % C H N O F
610.2549 610.2550 -0.1 -0.2 10.5 C21 H30 N13 O2 F6 567.0 7.162 0.08 21 30 13 2 6
610.2547 0.2 0.3 14.5 C19 H27 N19 O2 F3 568.2 8.316 0.02 19 27 19 2 3
610.2551 -0.2 -0.3 21.5 C29 H29 N13 O2 F 568.0 8.163 0.03 29 29 13 2 1
610.2546 0.3 0.5 3.5 C11 H28 N19 O2 F8 571.4 11.534 0.00 11 28 19 2 8
610.2549 -0.3 -0.5 6.5 C23 H33 N7 O2 F9 565.4 5.574 0.38 23 33 7 2 9
JP-78_09042019_002 484 (4.213) AM2 (Ar,30000.0,556.27,0.00,LS 1)
1: TOF MS ES+
610.2549
611.2588
2.23e+007
Ph
Ph
Ph
Ph
F
HN
O
3ea
For Help, press F1
10:18 AM
4/22/2019


7.6660
7.6498
7.2604
7.2328
7.0519
7.0330
7.0143
6.9986
6.9842
6.9740
6.9583
6.9438
6.8691
6.8621
6.8587
6.8374
6.8312
6.8144
6.8072
6.7995
6.7956
6.7745
6.7219
6.7127
6.6983
6.6824
6.6678
6.6444
6.6350
1.9092
0.0078
Ph
Ph
Ph
Ph
S
HN
O
3fa
1.00
1.01
10.12
6.41
4.44
5.34
1.01
3.02
f1 (ppm)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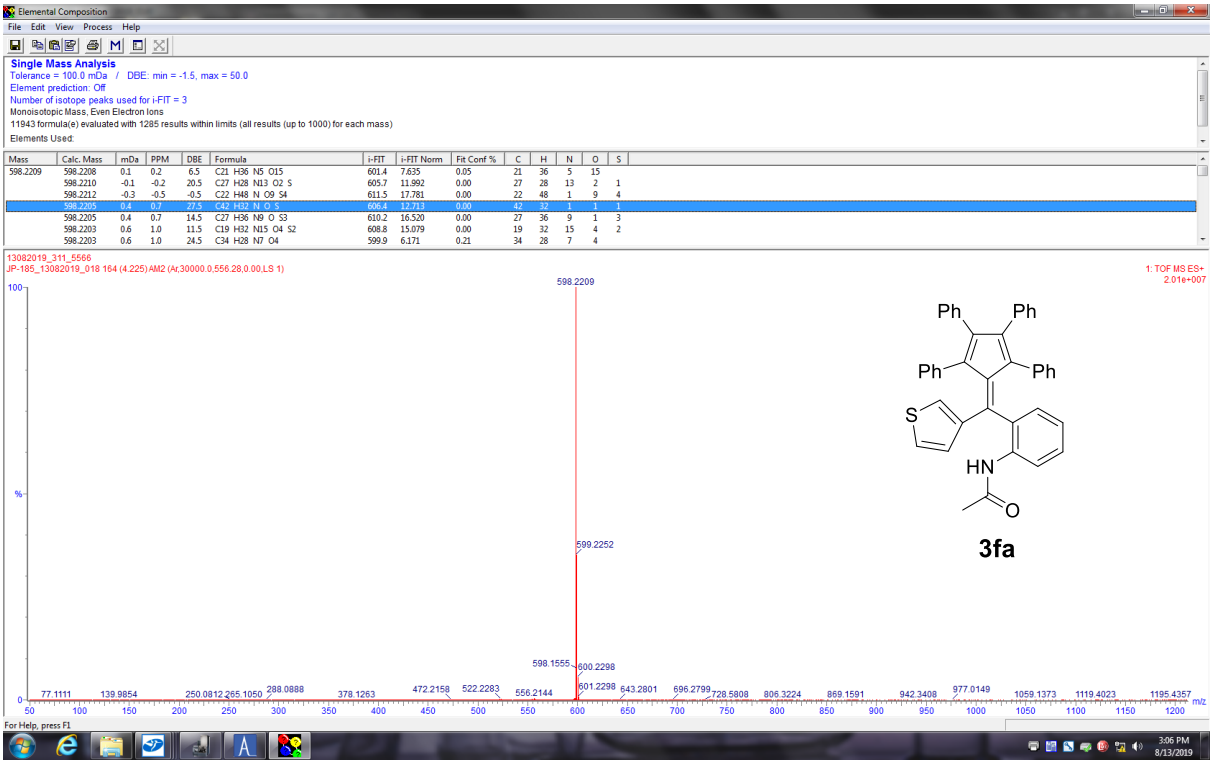
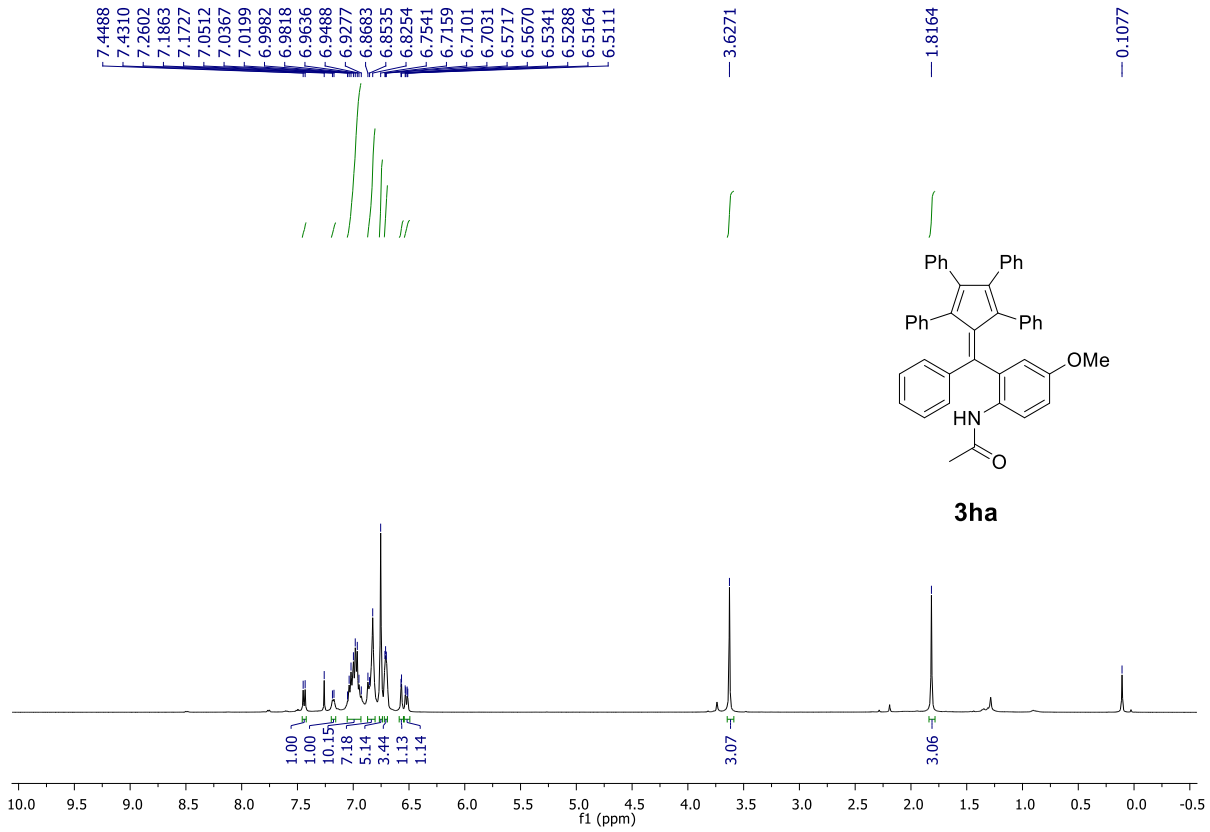
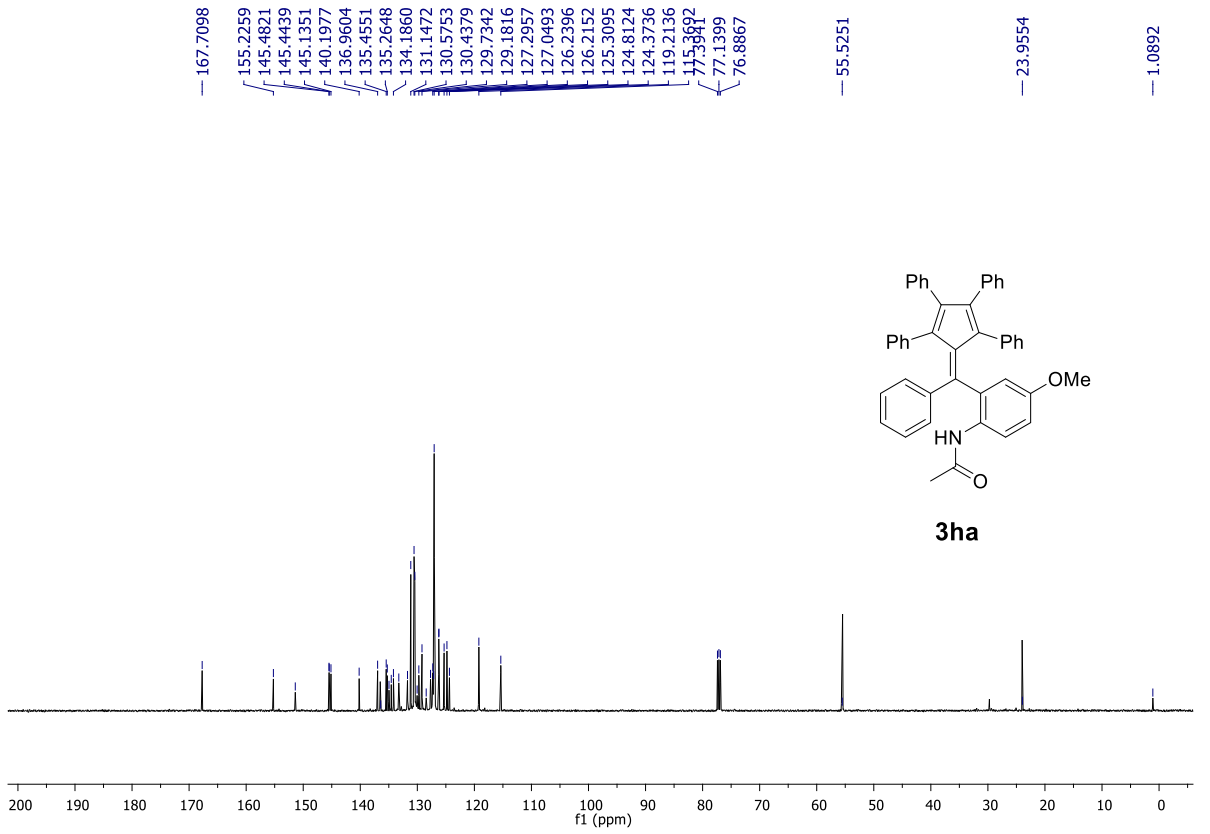
144.7536
141.9399
136.8927
135.3598
135.1130
133.5046
133.4841
131.0321
130.7410
130.4462
130.2478
130.1455
129.8711
127.0803
126.9623
126.9409
126.9211
126.0840
125.2008
124.9184
124.7234
123.2090
77.1918
76.9375
76.6830
24.2726
Ph
Ph
Ph
Ph
S
HN
O
3fa
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


Elemental Composition
File Edit View Process Help
Single Mass Analysis
Tolerance = 100.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3
Monoisotopic Mass, Even Electron Ions
11943 formula(e) evaluated with 1285 results within limits (all results (up to 1000) for each mass)
Elements Used:
Mass | Calc. Mass | mDa | PPM | DBE | Formula | i-FIT | i-FIT Norm | Fit Conf % | C | H | N | O | S
598.2209 | 598.2208 | 0.1 | 0.2 | 6.5 | C21 H36 N5 O15 | 601.4 | 7.635 | 0.05 | 21 | 36 | 5 | 15
598.2210 | -0.1 | -0.2 | 20.5 | C27 H28 N13 O2 S | 605.7 | 11.992 | 0.00 | 27 | 28 | 13 | 2 | 1
598.2212 | -0.3 | -0.5 | -0.5 | C22 H48 N O9 S4 | 611.5 | 17.781 | 0.00 | 22 | 48 | 1 | 9 | 4
598.2205 | 0.4 | 0.7 | 27.5 | C42 H32 N O S | 606.4 | 12.713 | 0.00 | 42 | 32 | 1 | 1 | 1
598.2205 | 0.4 | 0.7 | 14.5 | C27 H36 N9 O S3 | 610.2 | 16.520 | 0.00 | 27 | 36 | 9 | 1 | 3
598.2203 | 0.6 | 1.0 | 11.5 | C19 H32 N15 O4 S2 | 608.8 | 15.079 | 0.00 | 19 | 32 | 15 | 4 | 2
598.2203 | 0.6 | 1.0 | 24.5 | C34 H28 N7 O4 | 599.9 | 6.171 | 0.21 | 34 | 28 | 7 | 4
13082019_311_5566
JP-185_13082019_018 164 (4.225) AM2 (Ar,30000.0,556.28,0.00,LS 1)
1: TOF MS ES+
2.01e+007
100
%
0
598.2209
599.2252
598.1555
600.2298
601.2298
77.1111
139.9854
250.0812
265.1050
288.0888
378.1263
472.2158
522.2283
556.2144
643.2801
696.2799
728.5808
806.3224
869.1591
942.3408
977.0149
1059.1373
1119.4023
1195.4357
m/z
50
100
150
200
250
300
350
400
450
500
550
600
650
700
750
800
850
900
950
1000
1050
1100
1150
1200
Ph
Ph
Ph
Ph
S
HN
O
3fa
For Help, press F1
3:06 PM
8/13/2019


7.4488
7.4310
7.2602
7.1863
7.1727
7.0512
7.0367
7.0199
6.9982
6.9818
6.9636
6.9488
6.9277
6.8683
6.8535
6.8254
6.7541
6.7159
6.7101
6.7031
6.5717
6.5670
6.5341
6.5288
6.5164
6.5111
3.6271
1.8164
0.1077
Ph
Ph
Ph
Ph
OMe
HN
O
1.00
1.00
10.15
7.18
5.14
3.44
1.13
1.14
3.07
3.06
f1 (ppm)

3ha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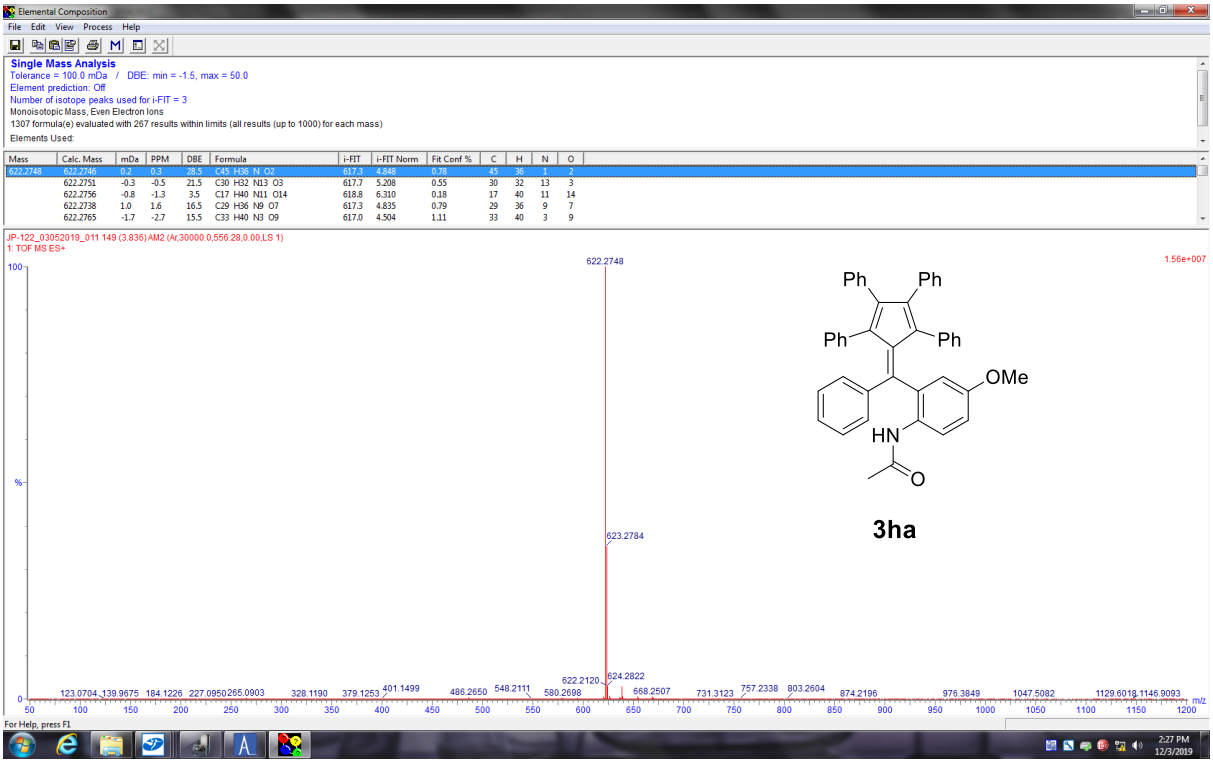
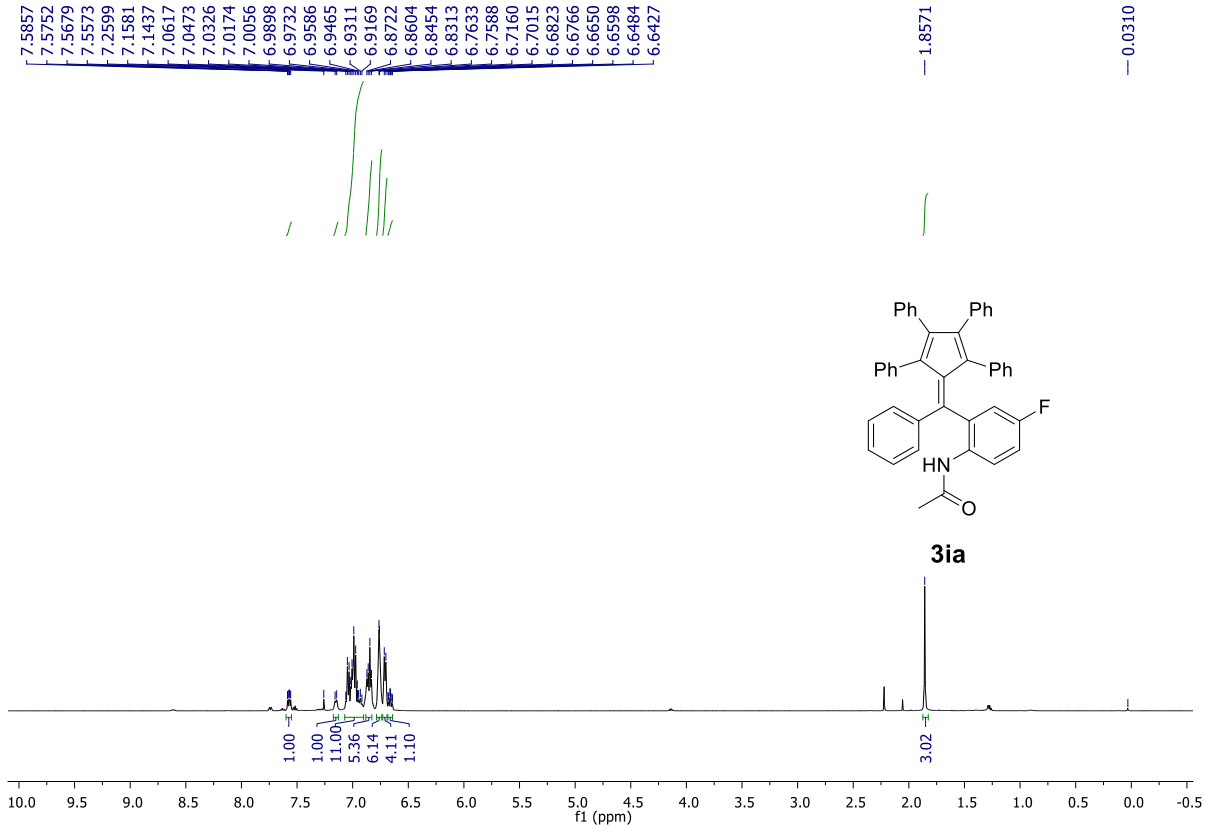
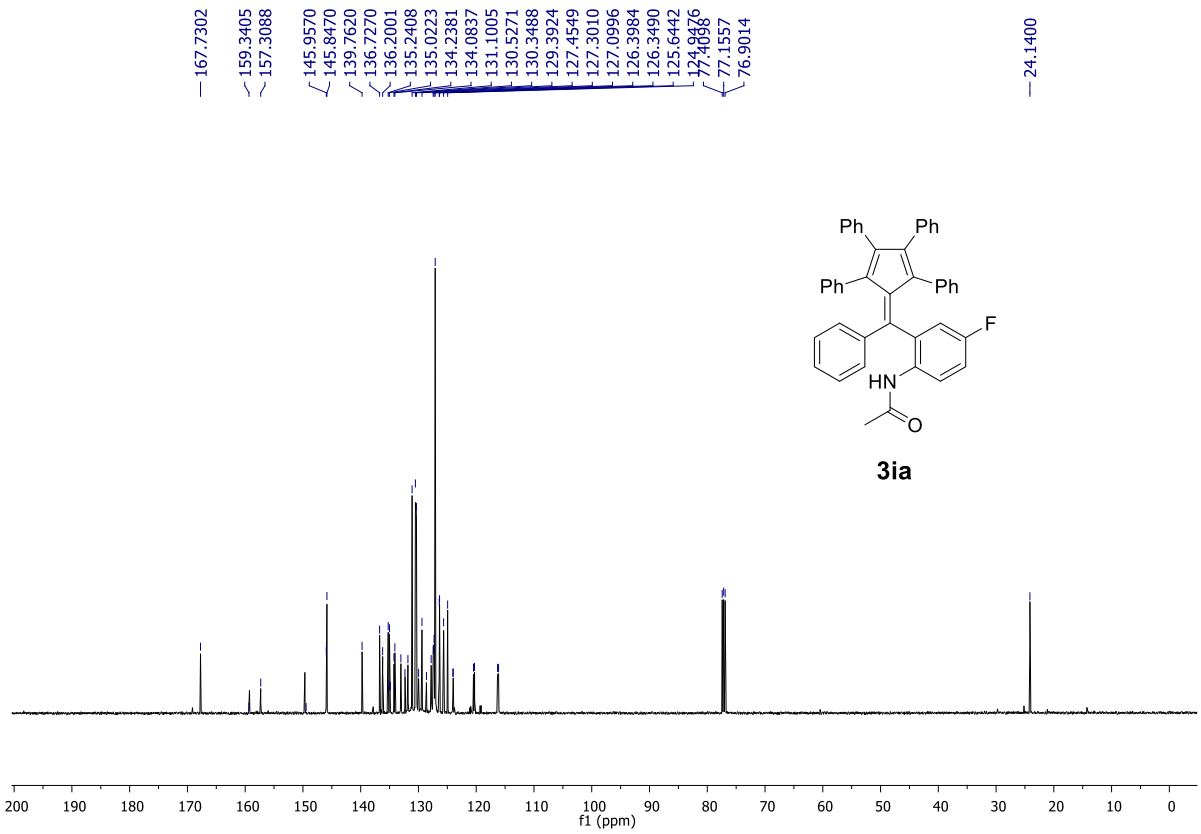
155.2259
145.4821
145.4439
145.1351
140.1977
136.9604
135.4551
135.2648
134.1860
131.1472
130.5753
130.4379
129.7342
129.1816
127.2957
127.0493
126.2396
126.2152
125.3095
124.8124
124.3736
119.2136
77.3911
77.1399
76.8867
55.5251
23.9554
1.0892
Ph
Ph
Ph
Ph
OMe
HN
O
f1 (ppm)

3ha


Elemental Composition
Single Mass Analysis
Tolerance = 100.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3
Monoisotopic Mass, Even Electron Ions
1307 formula(e) evaluated with 267 results within limits (all results (up to 1000) for each mass)
Elements Used:
622.2748
623.2784
622.2120
624.2822
Ph
Ph
Ph
Ph
OMe
HN
O
3ha


7.5857
7.5752
7.5679
7.5573
7.2599
7.1581
7.1437
7.0617
7.0473
7.0326
7.0174
7.0056
6.9898
6.9732
6.9586
6.9465
6.9311
6.9169
6.8722
6.8604
6.8454
6.8313
6.7633
6.7588
6.7160
6.7015
6.6823
6.6766
6.6650
6.6598
6.6484
6.6427
1.8571
0.0310
Ph
Ph
Ph
Ph
F
HN
O
3ia
1.00
1.00
11.00
5.36
6.14
4.11
1.10
3.02
f1 (ppm)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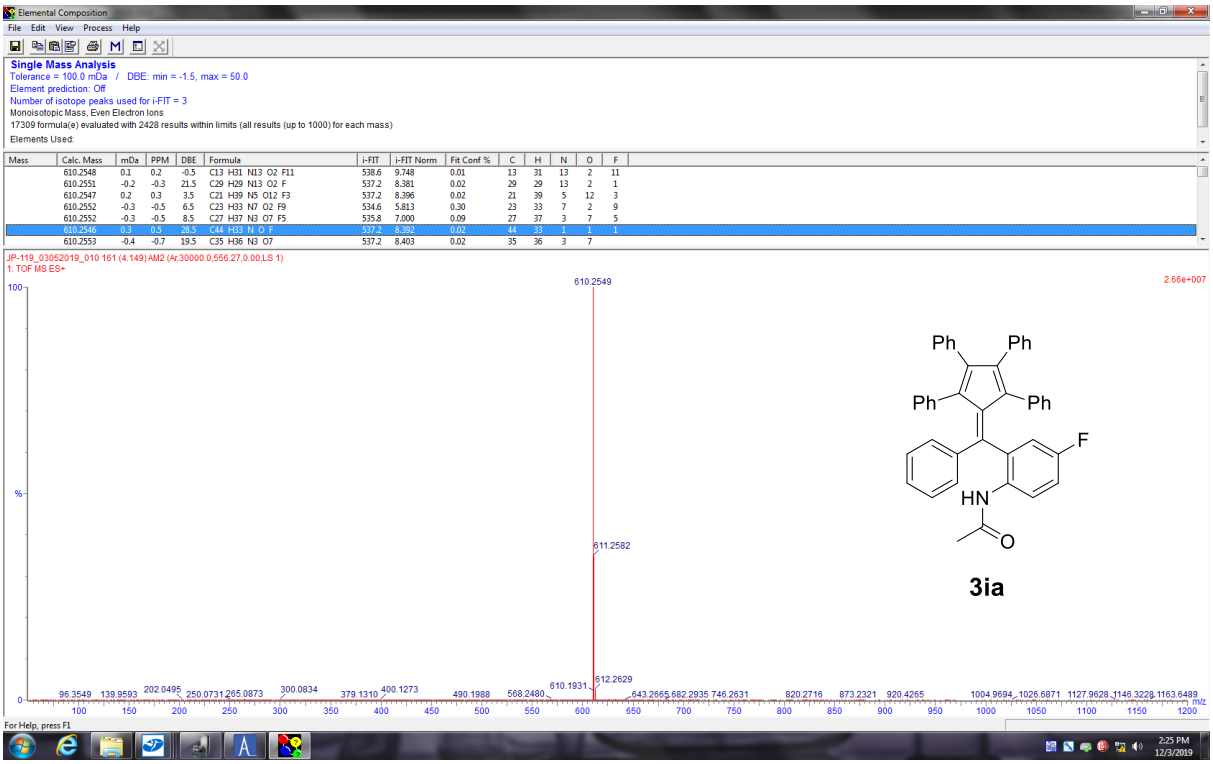
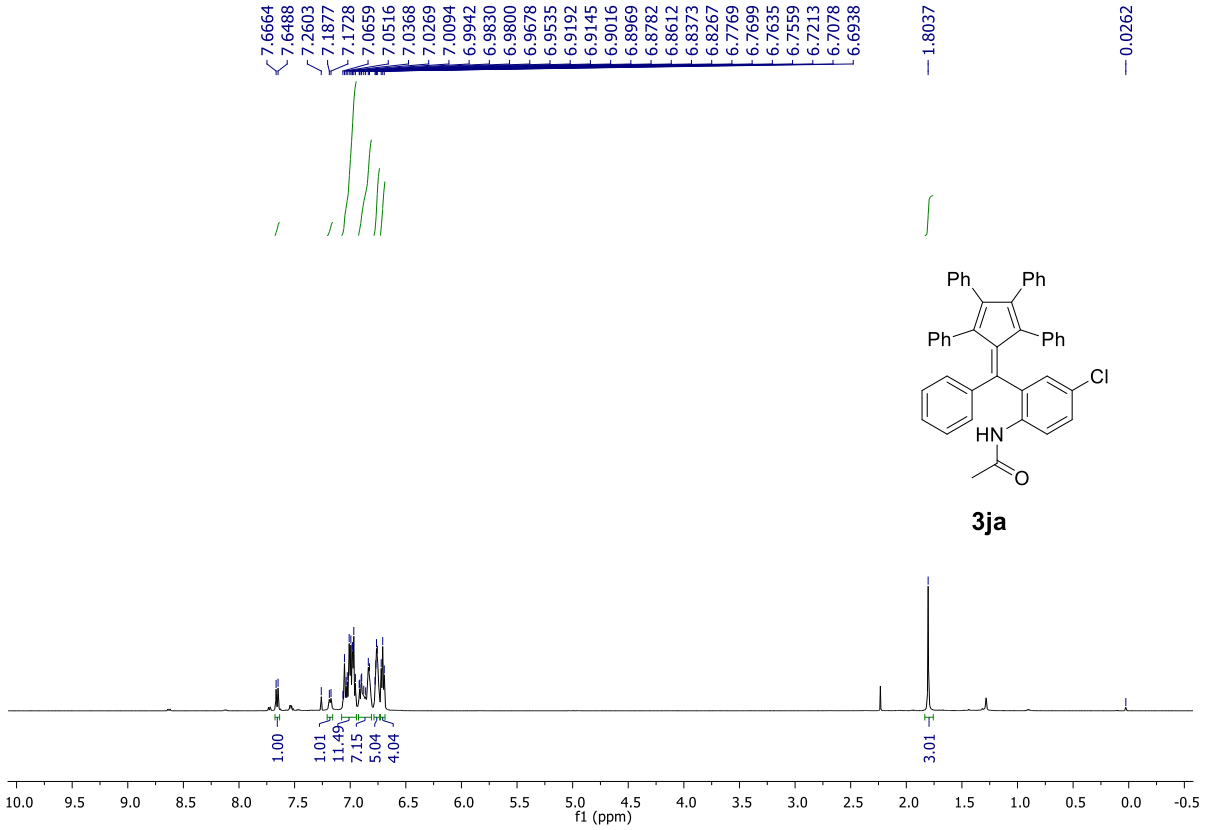
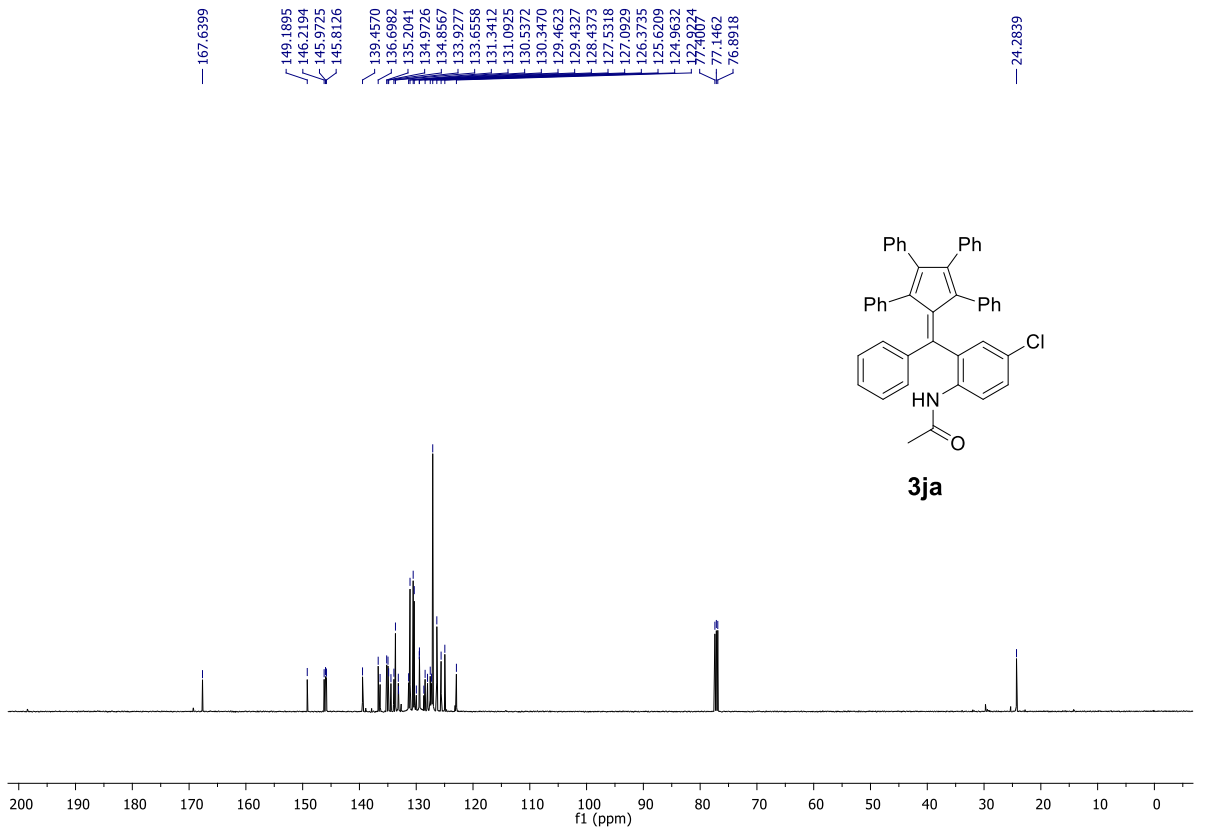
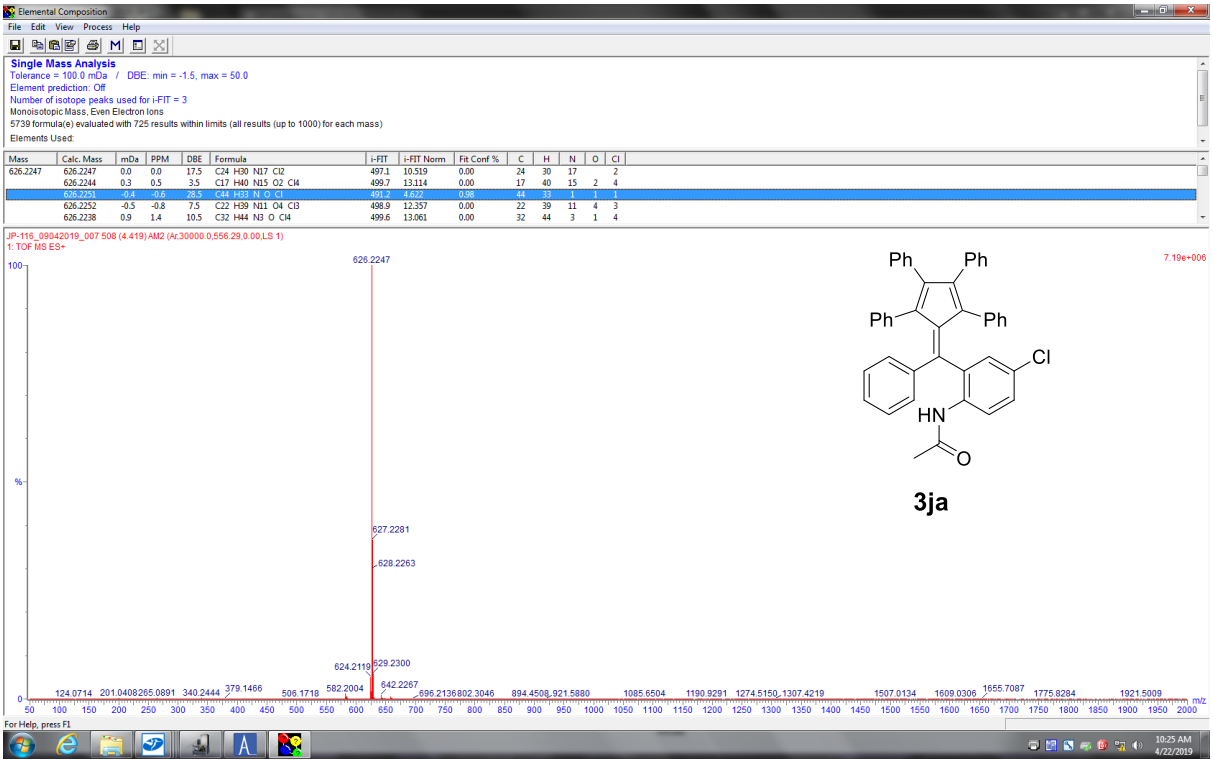
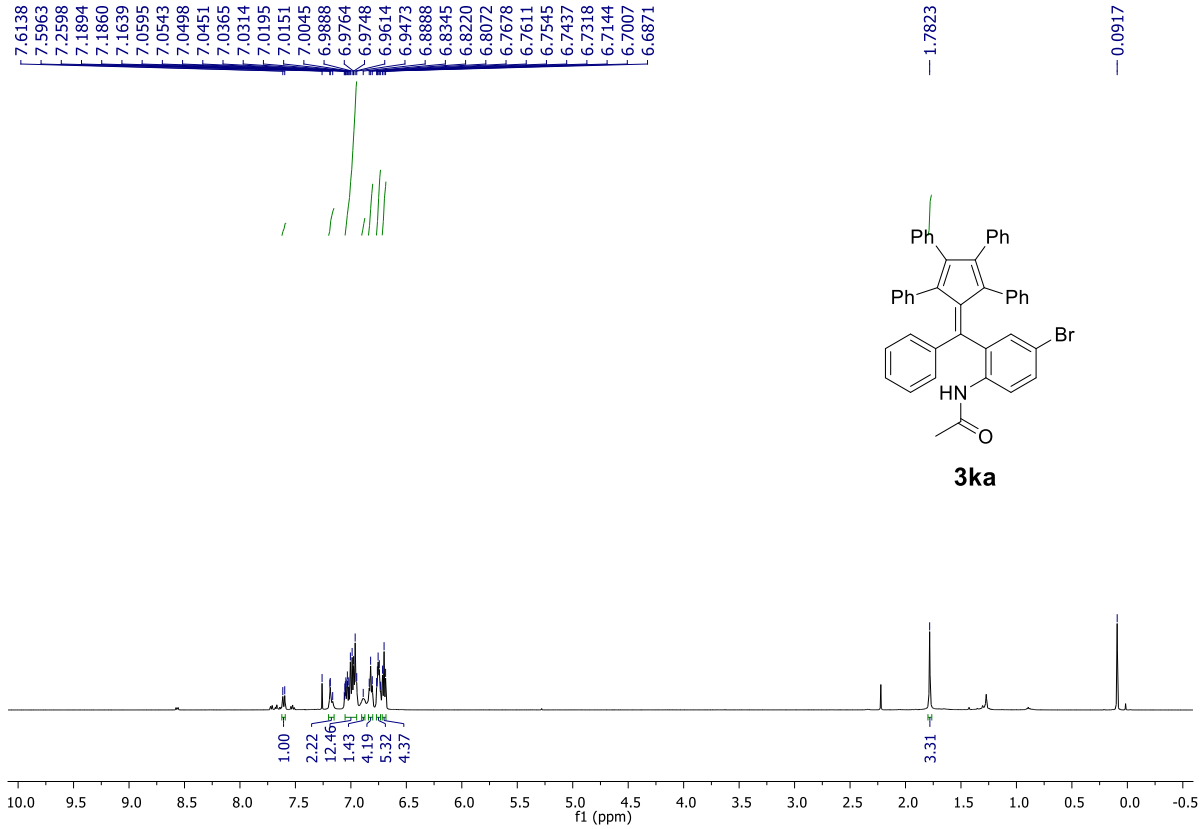
159.3405
157.3088
145.9570
145.8470
139.7620
136.7270
136.2001
135.2408
135.0223
134.2381
134.0837
131.1005
130.5271
130.3488
129.3924
127.4549
127.3010
127.0996
126.3984
126.3490
125.6442
124.9476
77.4098
77.1557
76.9014
24.1400
Ph
Ph
Ph
Ph
F
HN
O
3ia
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

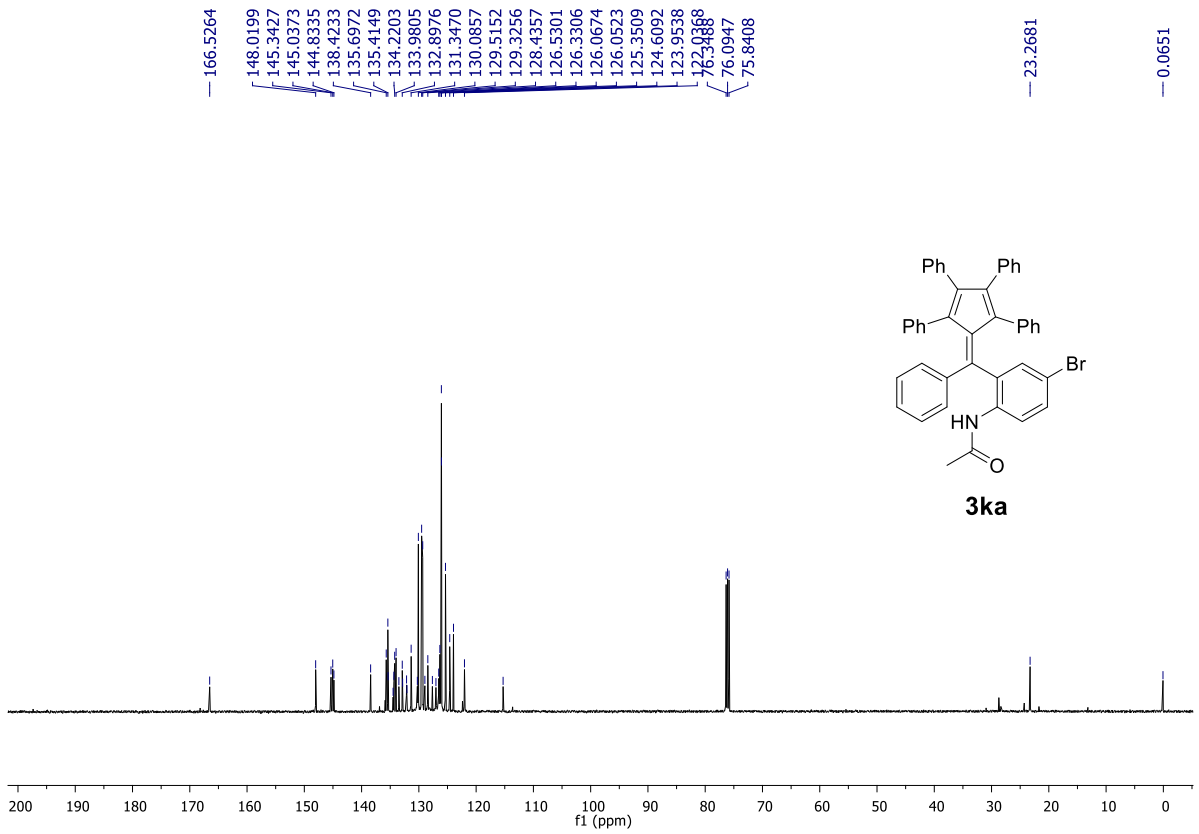

Elemental Composition
File Edit View Process Help
Single Mass Analysis
Tolerance = 100.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3
Monoisotopic Mass, Even Electron Ions
17309 formula(e) evaluated with 2428 results within limits (all results (up to 1000) for each mass)
Elements Used:
Mass | Calc. Mass | mDa | PPM | DBE | Formula | i-FIT | i-FIT Norm | Fit Conf % | C | H | N | O | F
610.2548 0.1 0.2 -0.5 C13 H31 N13 O2 F11 538.6 9.748 0.01 13 31 13 2 11
610.2551 -0.2 -0.3 21.5 C29 H29 N13 O2 F 537.2 8.381 0.02 29 29 13 2 1
610.2547 0.2 0.3 3.5 C21 H39 N5 O12 F3 537.2 8.396 0.02 21 39 5 12 3
610.2552 -0.3 -0.5 6.5 C23 H33 N7 O2 F9 534.6 5.813 0.30 23 33 7 2 9
610.2552 -0.3 -0.5 8.5 C27 H37 N3 O7 F5 535.8 7.000 0.09 27 37 3 7 5
610.2546 0.3 0.5 28.5 C44 H33 N O F 537.2 8.392 0.02 44 33 1 1 1
610.2553 -0.4 -0.7 19.5 C35 H36 N3 O7 537.2 8.403 0.02 35 36 3 7
JP-119_03052019_010 161 (4.149) AM2 (Ar,30000.0,556.27,0.00,LS 1)
1: TOF MS ES+
610.2549
2.66e+007
611.2582
Ph
Ph
Ph
Ph
F
HN
O
3ia
For Help, press F1
2:25 PM
12/3/2019

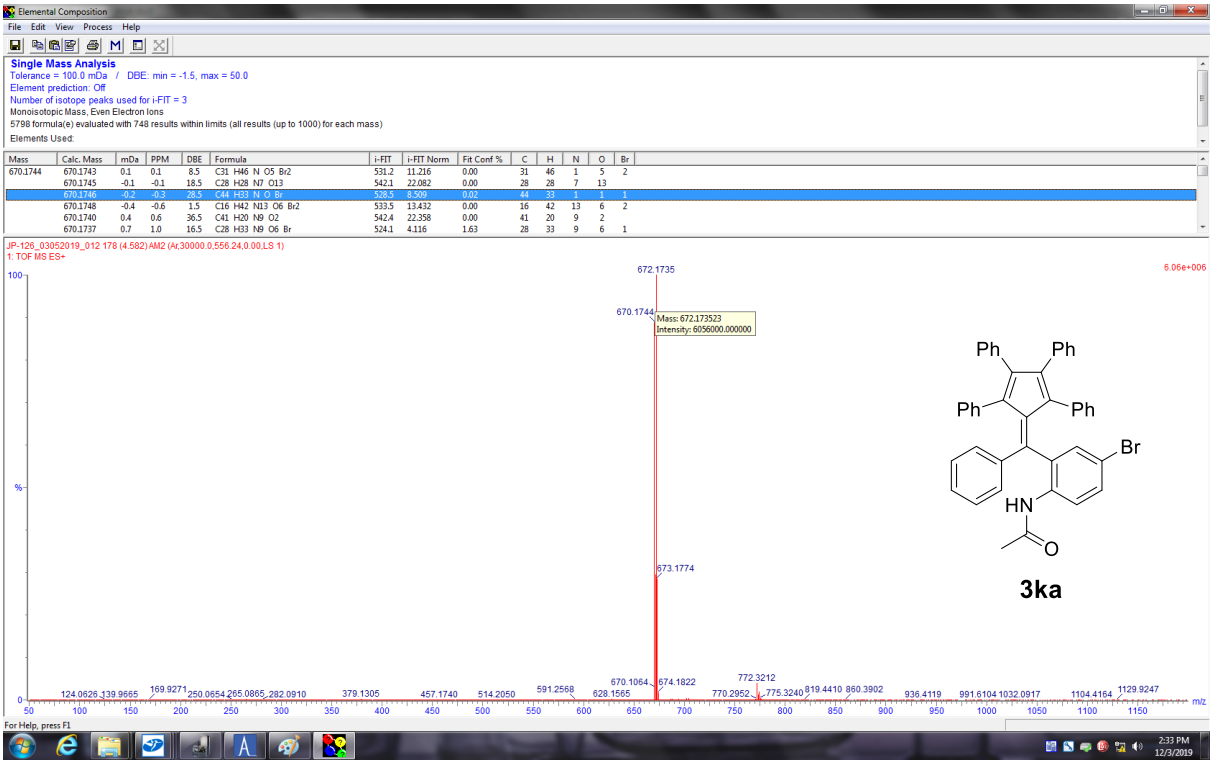

7.6664
7.6488
7.2603
7.1877
7.1728
7.0659
7.0516
7.0368
7.0269
7.0094
6.9942
6.9830
6.9800
6.9678
6.9535
6.9192
6.9145
6.9016
6.8969
6.8782
6.8612
6.8373
6.8267
6.7769
6.7699
6.7635
6.7559
6.7213
6.7078
6.6938
1.8037
0.0262
Ph
Ph
Ph
Ph
Cl
HN
O
3ja
1.00
1.01
11.49
7.15
5.04
4.04
3.01
f1 (ppm)

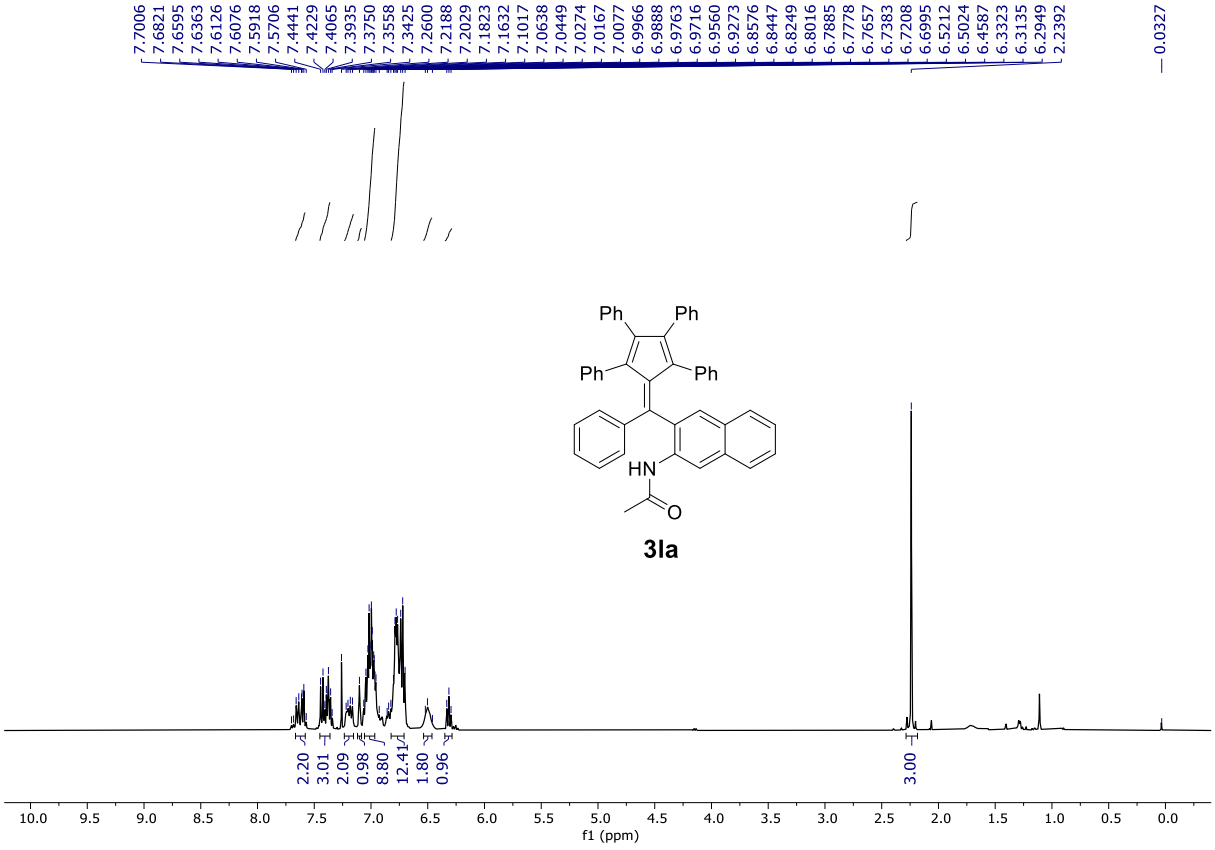

167.6399
149.1895
146.2194
145.9725
145.8126
139.4570
136.6982
135.2041
134.9726
134.8567
133.9277
133.6558
131.3412
131.0925
130.5372
130.3470
129.4623
129.4327
128.4373
127.5318
127.0929
126.3735
125.6209
124.9632
77.1462
76.8918
24.2839
Ph
Ph
Ph
Ph
Cl
HN
O
3ja
f1 (ppm)

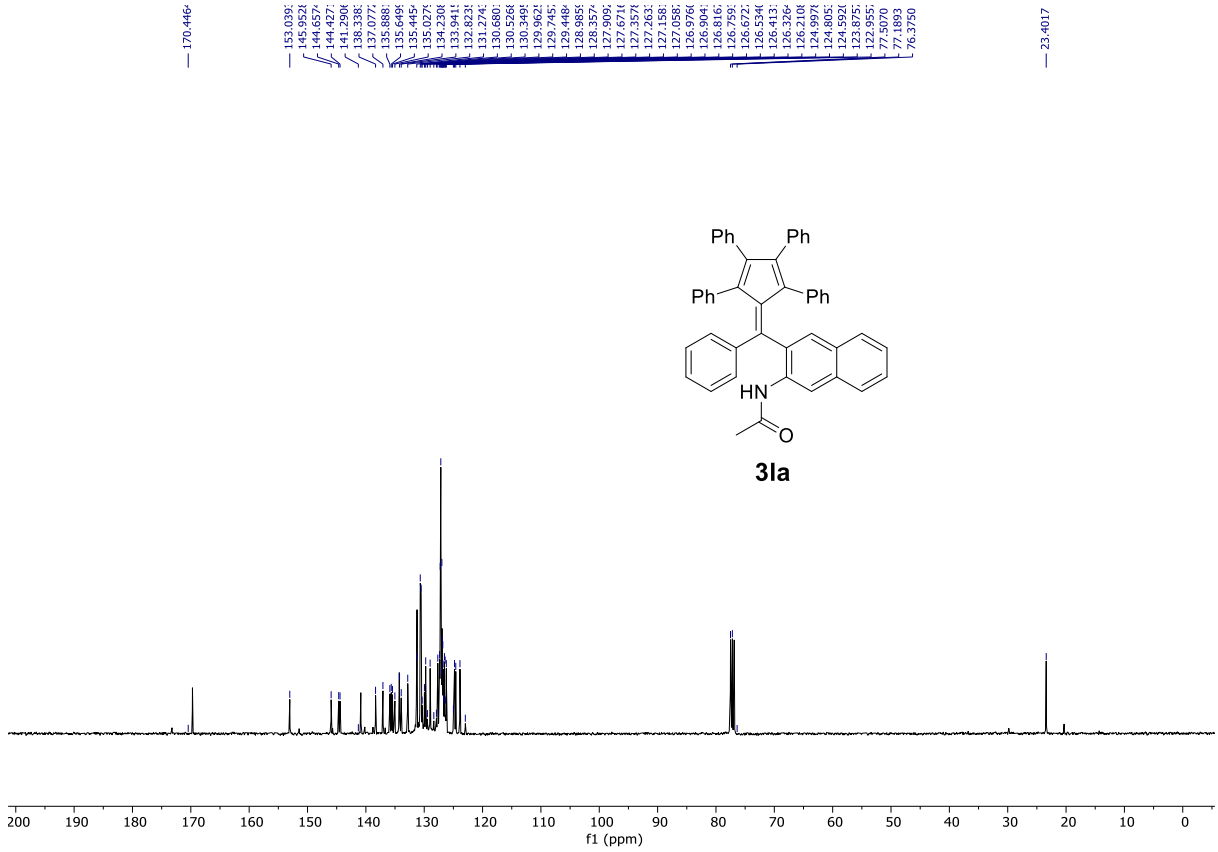

Elemental Composition
Single Mass Analysis
Tolerance = 100.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3
Monoisotopic Mass, Even Electron Ions
5739 formula(e) evaluated with 725 results within limits (all results (up to 1000) for each mass)
Elements Used:
626.2247
627.2281
628.2263
629.2300
624.2119
642.2267
582.2004
Ph
Ph
Ph
Ph
Cl
HN
O
3ja

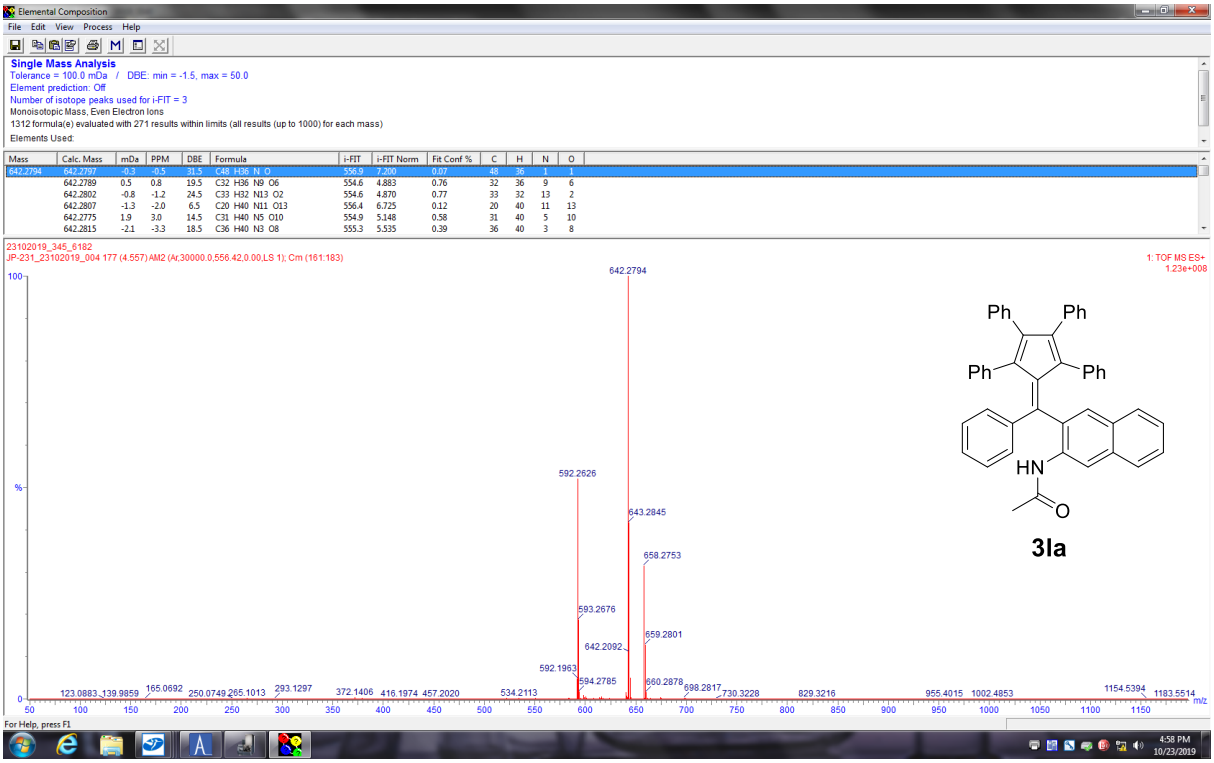

7.6138
7.5963
7.2598
7.1894
7.1860
7.1639
7.0595
7.0543
7.0498
7.0451
7.0365
7.0314
7.0195
7.0151
7.0045
6.9888
6.9764
6.9748
6.9614
6.9473
6.8888
6.8345
6.8220
6.8072
6.7678
6.7611
6.7545
6.7437
6.7318
6.7144
6.7007
6.6871
1.7823
0.0917
Ph
Ph
Ph
Ph
Br
HN
O
3ka
1.00
2.22
12.46
1.43
4.19
5.32
4.37
3.31
f1 (ppm)

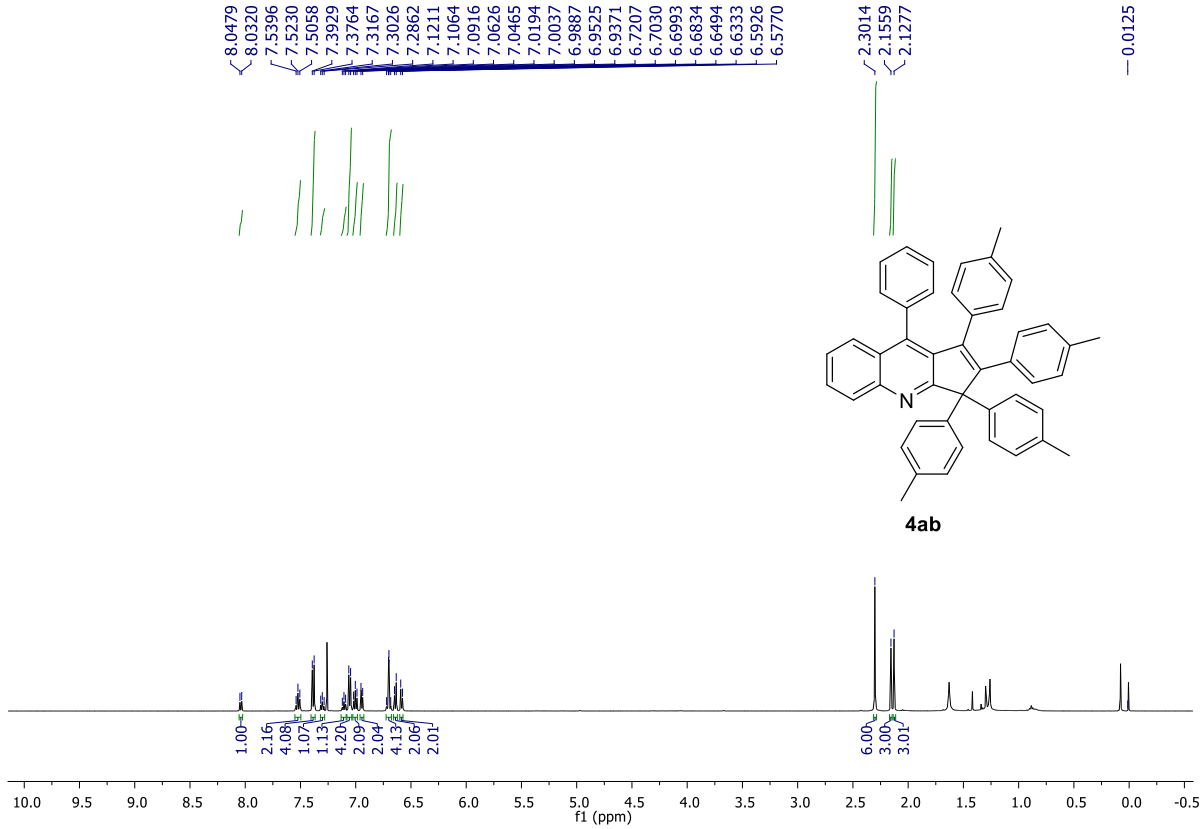

166.5264
148.0199
145.3427
145.0373
144.8335
138.4233
135.6972
135.4149
134.2203
133.9805
132.8976
131.3470
130.0857
129.5152
129.3256
128.4357
126.5301
126.3306
126.0674
126.0523
125.3509
124.6092
123.9538
122.0368
76.0947
75.8408
23.2681
0.0651
Ph
Ph
Ph
Ph
Br
HN
O
3ka
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

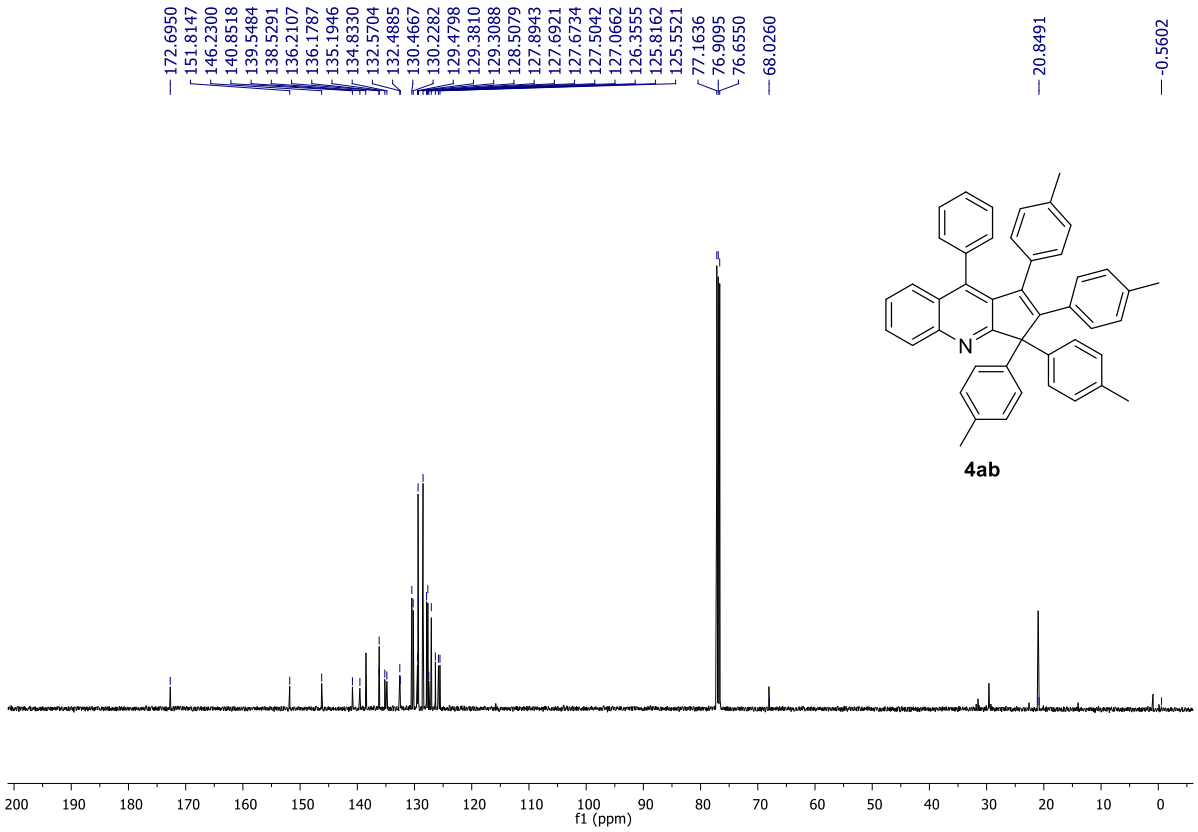

Elemental Composition
File Edit View Process Help
Single Mass Analysis
Tolerance = 100.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3
Monoisotopic Mass, Even Electron Ions
5798 formula(e) evaluated with 748 results within limits (all results (up to 1000) for each mass)
Elements Used:
Mass Calc. Mass mDa PPM DBE Formula i-FIT i-FIT Norm Fit Conf % C H N O Br
670.1744 670.1743 0.1 0.1 8.5 C31 H46 N O5 Br2 531.2 11.216 0.00 31 46 1 5 2
670.1745 -0.1 -0.1 18.5 C28 H28 N7 O13 542.1 22.082 0.00 28 28 7 13
670.1746 -0.2 -0.3 28.5 C44 H33 N O Br 528.5 8.509 0.02 44 33 1 1 1
670.1748 -0.4 -0.6 1.5 C16 H42 N13 O6 Br2 533.5 13.432 0.00 16 42 13 6 2
670.1740 0.4 0.6 36.5 C41 H20 N9 O2 542.4 22.358 0.00 41 20 9 2
670.1737 0.7 1.0 16.5 C28 H33 N9 O6 Br 524.1 4.116 1.63 28 33 9 6 1
JP-126_03052019_012 178 (4.582) AM2 (Ar,30000.0,556.24,0.00,LS 1)
1: TOF MS ES+
6.06e+006
672.1735
670.1744
Mass: 672.173523
Intensity: 6056000.000000
673.1774
Ph
Ph
Ph
Ph
Br
HN
O
3ka
For Help, press F1
2:33 PM
12/3/2019

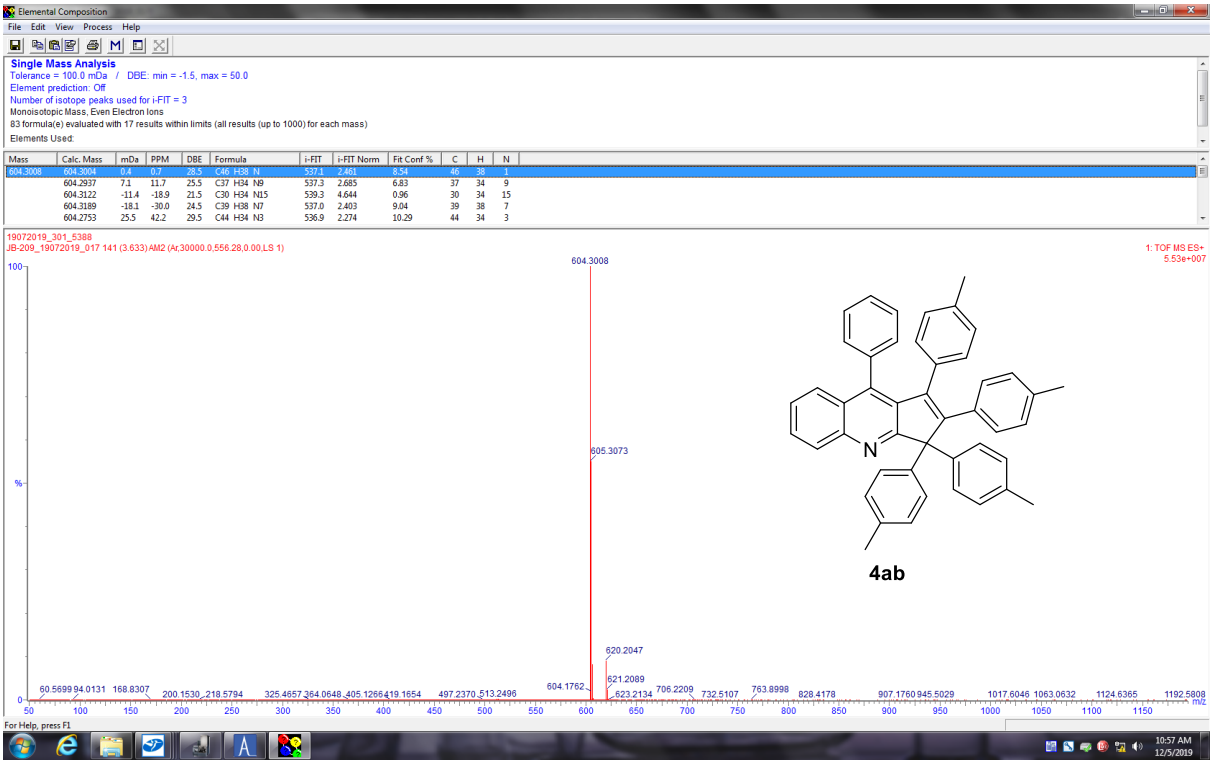

7.7006
7.6821
7.6595
7.6363
7.6126
7.6076
7.5918
7.5706
7.4441
7.4229
7.4065
7.3935
7.3750
7.3558
7.3425
7.2600
7.2188
7.2029
7.1823
7.1632
7.1017
7.0638
7.0449
7.0274
7.0167
7.0077
6.9966
6.9888
6.9763
6.9716
6.9560
6.9273
6.8576
6.8447
6.8249
6.8016
6.7885
6.7778
6.7657
6.7383
6.7208
6.6995
6.5212
6.5024
6.4587
6.3323
6.3135
6.2949
6.2392
0.0327
Ph
Ph
Ph
Ph
HN
O
3Ia
2.20
3.01
2.09
0.98
8.80
12.41
1.80
0.96
3.00
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)

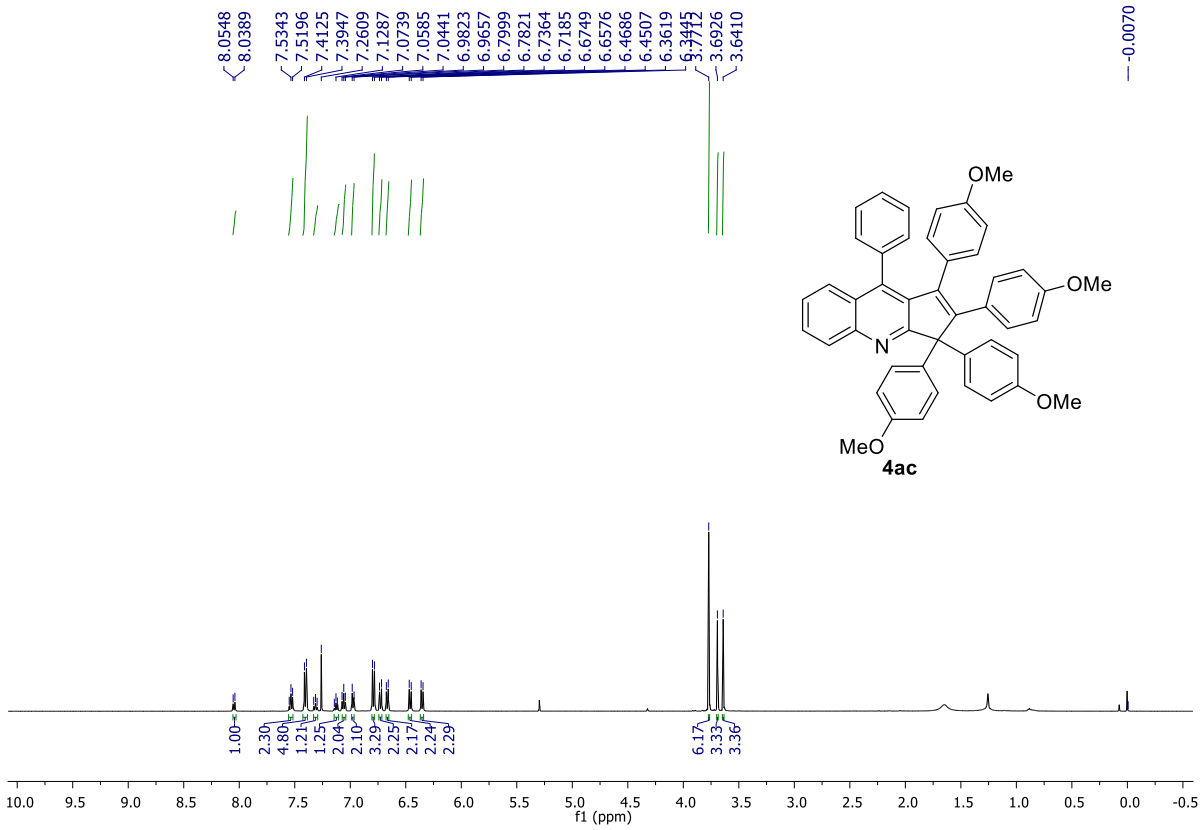

170.446
153.039
145.952
144.657
141.427
138.290
137.077
135.888
135.649
135.445
135.027
134.230
133.941
132.823
131.274
130.680
130.526
130.349
129.962
129.745
129.448
128.985
128.357
127.909
127.671
127.357
127.263
127.158
127.058
126.976
126.904
126.816
126.759
126.672
126.534
126.413
126.326
126.210
124.997
124.805
124.592
123.875
122.955
77.5070
77.1893
76.3750
23.4017
Ph
Ph
Ph
Ph
HN
O
3Ia
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

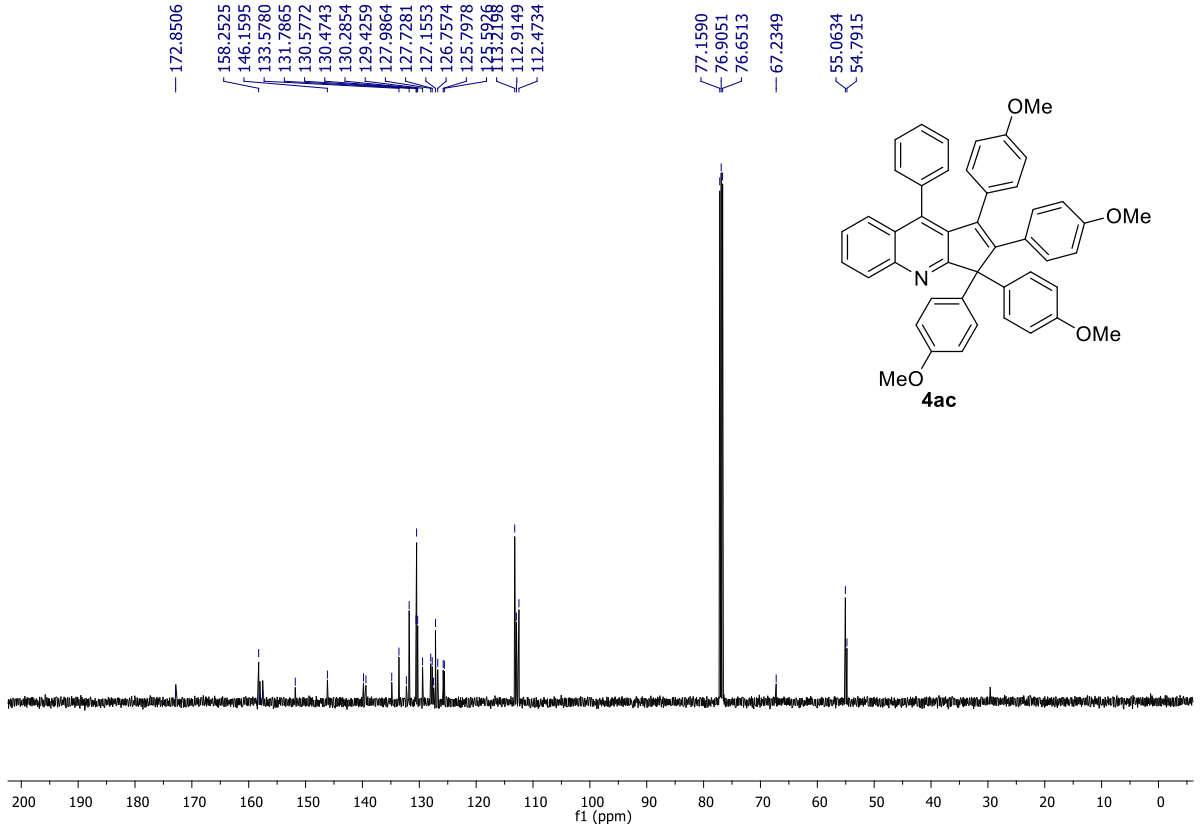

Elemental Composition
Single Mass Analysis
Tolerance = 100.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3
Monoisotopic Mass, Even Electron Ions
1312 formula(e) evaluated with 271 results within limits (all results (up to 1000) for each mass)
Elements Used:
642.2794
642.2626
643.2845
658.2753
593.2676
659.2801
642.2092
592.1963
594.2785
660.2878
Ph
Ph
Ph
Ph
HN
O
3la

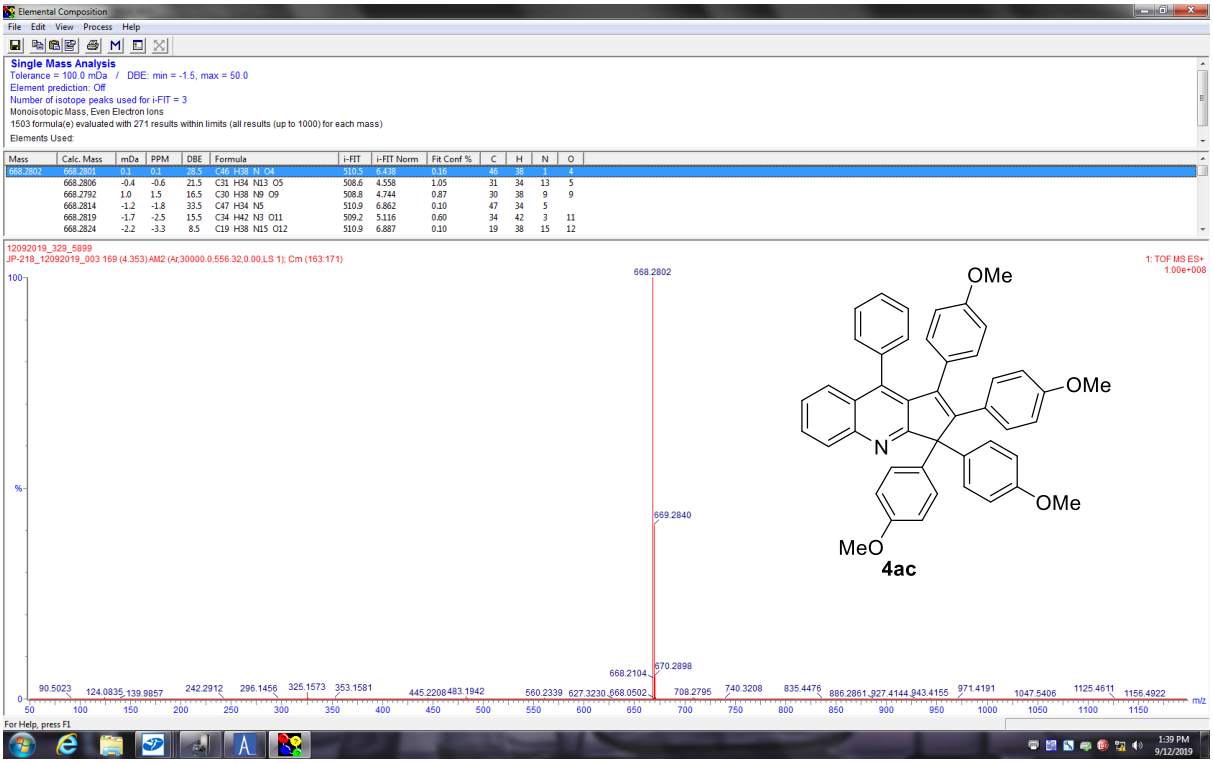

8.0479
8.0320
7.5396
7.5230
7.5058
7.3929
7.3764
7.3167
7.3026
7.2862
7.1211
7.1064
7.0916
7.0626
7.0465
7.0194
7.0037
6.9887
6.9525
6.9371
6.7207
6.7030
6.6993
6.6834
6.6494
6.6333
6.5926
6.5770
2.3014
2.1559
2.1277
0.0125
N
4ab
1.00
2.16
4.08
1.07
1.13
4.20
2.09
2.04
4.13
2.06
2.01
6.00
3.00
3.01
f1 (ppm)

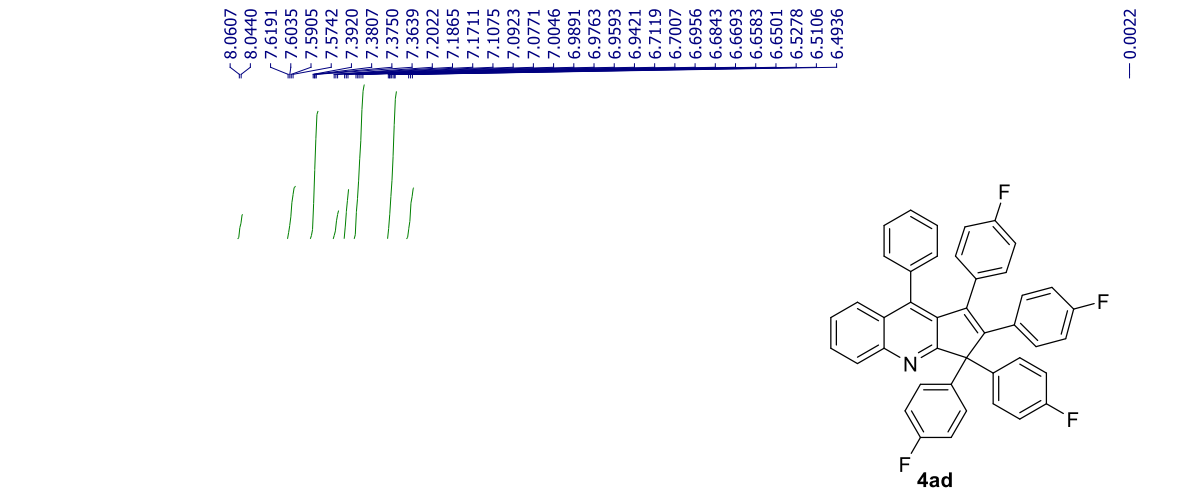

172.6950
151.8147
146.2300
140.8518
139.5484
138.5291
136.2107
136.1787
135.1946
134.8330
132.5704
132.4885
130.4667
130.2282
129.4798
129.3810
129.3088
128.5079
127.8943
127.6921
127.6734
127.5042
127.0662
126.3555
125.8162
125.5521
77.1636
76.9095
76.6550
68.0260
20.8491
-0.5602
N
4ab
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

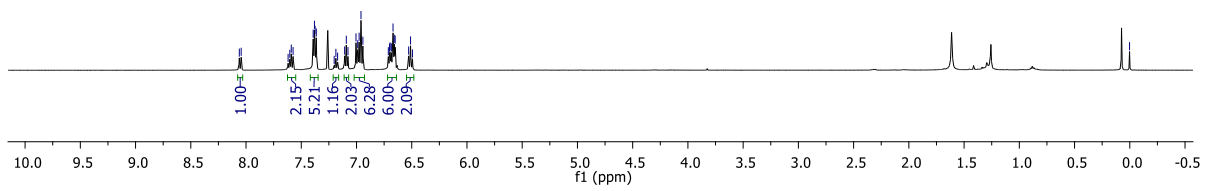

Elemental Composition
File Edit View Process Help
Single Mass Analysis
Tolerance = 100.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3
Monoisotopic Mass, Even Electron Ions
83 formula(e) evaluated with 17 results within limits (all results (up to 1000) for each mass)
Elements Used:
Mass Calc. Mass mDa PPM DBE Formula i-FIT i-FIT Norm Fit Conf % C H N
604.3008 604.3004 0.4 0.7 28.5 C46 H38 N 537.1 2.461 8.54 46 38 1
604.2937 7.1 11.7 25.5 C37 H34 N8 537.3 2.685 6.83 37 34 9
604.3122 -11.4 -18.9 21.5 C30 H34 N15 539.3 4.644 0.96 30 34 15
604.3189 -18.1 -30.0 24.5 C39 H38 N7 537.0 2.403 9.04 39 38 7
604.2753 25.5 42.2 29.5 C44 H34 N3 536.9 2.274 10.29 44 34 3
19072019_301_5388
JB-209_19072019_017 141 (3.633) AM2 (Ar,30000.0,556.28,0.00,LS 1)
1: TOF MS ES+
5.53e+007
604.3008
605.3073
620.2047
621.2089
623.2134
N
4ab
For Help, press F1
10:57 AM
12/5/2019

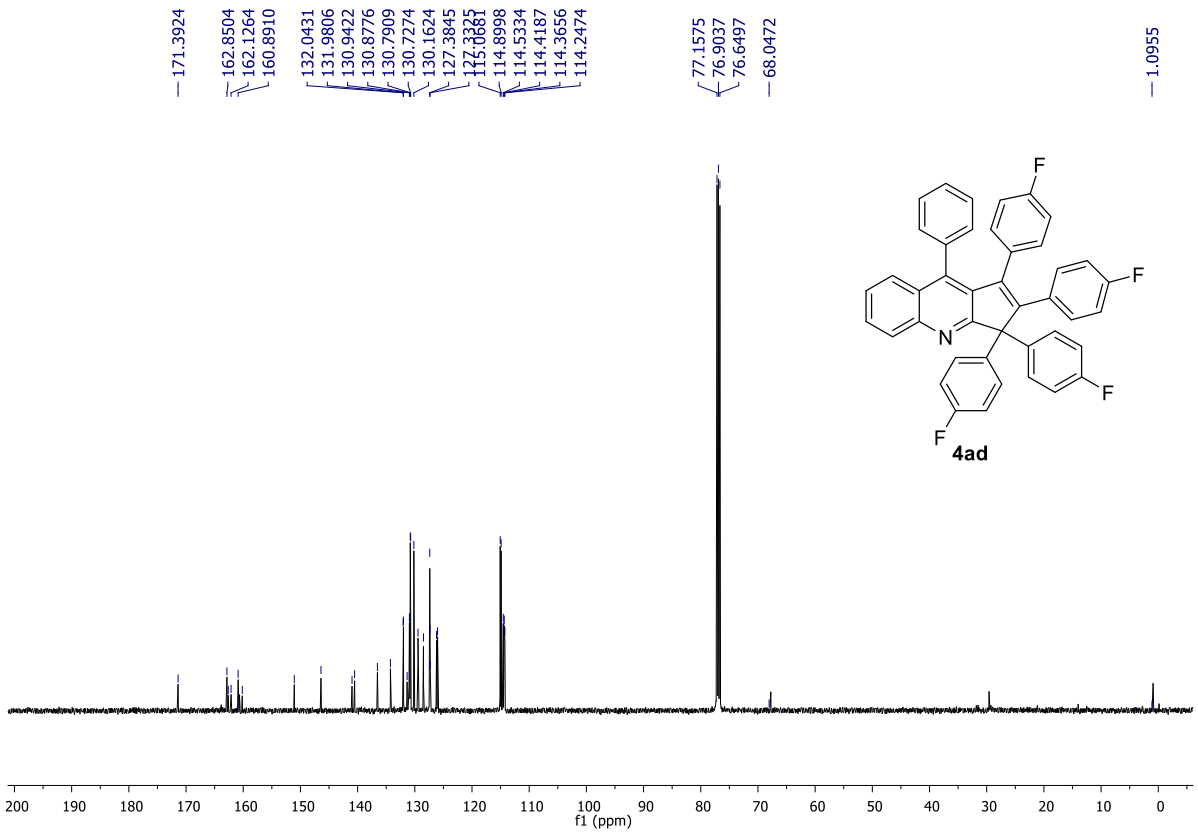

8.0548
8.0389
7.5343
7.5196
7.4125
7.3947
7.2609
7.1287
7.0739
7.0585
7.0441
6.9823
6.9657
6.7999
6.7821
6.7364
6.7185
6.6749
6.6576
6.4686
6.4507
6.3619
6.3445
3.7112
3.6926
3.6410
-0.0070
OMe
OMe
OMe
MeO
N
4ac
1.00
2.30
4.80
1.21
1.25
2.04
2.10
3.29
2.25
2.17
2.24
2.29
6.17
3.33
3.36
f1 (ppm)

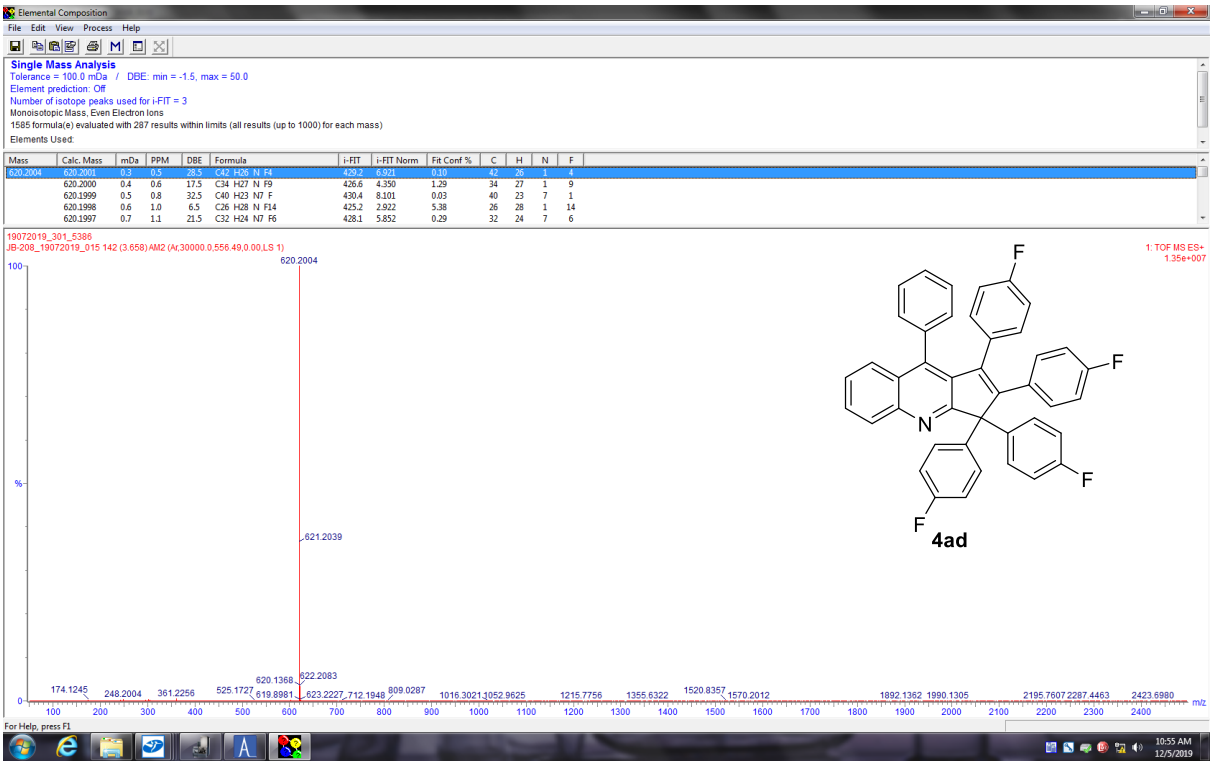

172.8506
158.2525
146.1595
133.5780
131.7865
130.5772
130.4743
130.2854
129.4259
127.9864
127.7281
127.1553
126.7574
125.7978
125.5926
113.2198
112.9149
112.4734
77.1590
76.9051
76.6513
67.2349
55.0634
54.7915
OMe
OMe
OMe
MeO
N
4ac
f1 (ppm)

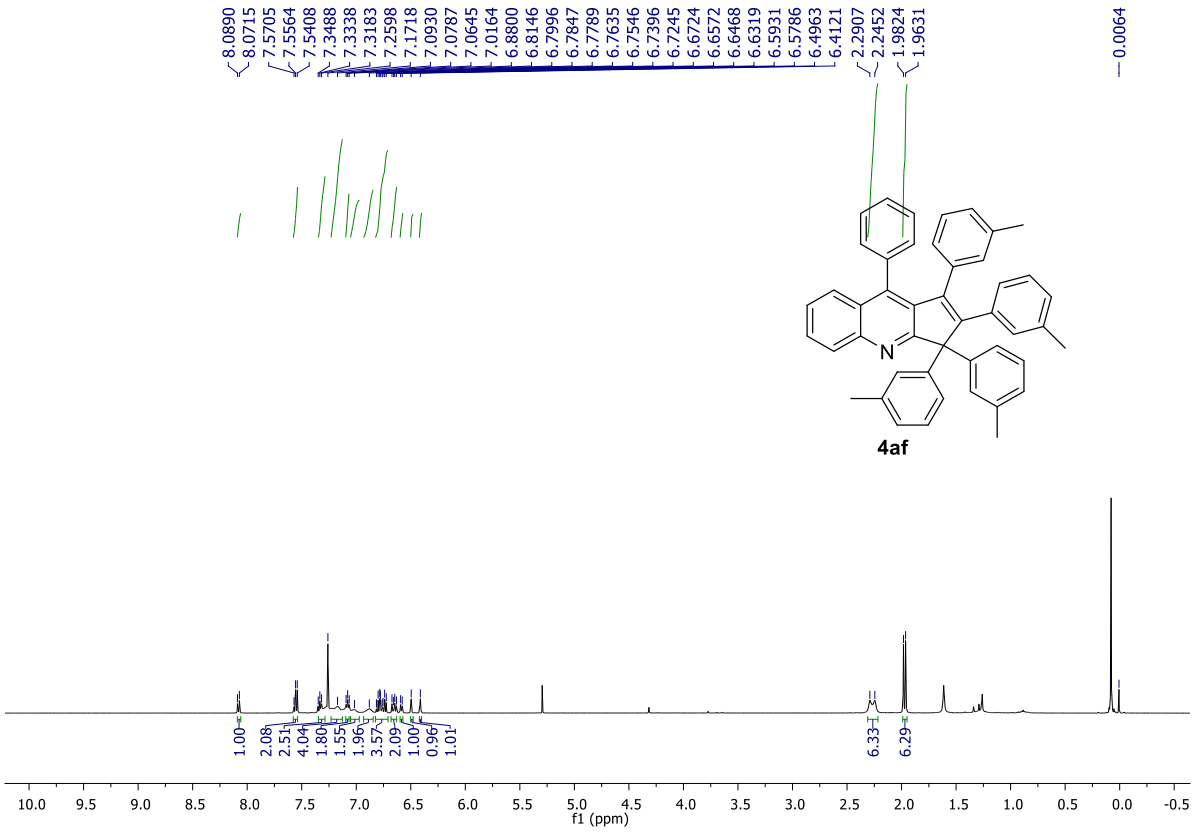

Elemental Composition
Single Mass Analysis
Tolerance = 100.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3
Monoisotopic Mass, Even Electron Ions
1503 formula(e) evaluated with 271 results within limits (all results (up to 1000) for each mass)
Elements Used:
668.2802
669.2840
670.2898
668.2104
OMe
OMe
OMe
MeO
N
4ac


8.0607
8.0440
7.6191
7.6035
7.5905
7.5742
7.3920
7.3807
7.3750
7.3639
7.2022
7.1865
7.1711
7.1075
7.0923
7.0771
7.0046
6.9891
6.9763
6.9593
6.9421
6.7119
6.7007
6.6956
6.6843
6.6693
6.6583
6.6501
6.5278
6.5106
6.4936
0.0022
F
F
F
F
N
4ad


1.00
2.15
5.21
1.16
2.03
6.28
6.00
2.09
f1 (ppm)


171.3924
162.8504
162.1264
160.8910
132.0431
131.9806
130.9422
130.8776
130.7909
130.7274
130.1624
127.3845
127.3325
115.0681
114.8998
114.5334
114.4187
114.3656
114.2474
77.1575
76.9037
76.6497
68.0472
1.0955
F
F
F
F
N
4ad
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


Elemental Composition
File Edit View Process Help
Single Mass Analysis
Tolerance = 100.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3
Monoisotopic Mass, Even Electron Ions
1585 formula(e) evaluated with 287 results within limits (all results (up to 1000) for each mass)
Elements Used:
Mass Calc. Mass mDa PPM DBE Formula i-FIT i-FIT Norm Fit Conf % C H N F
620.2004 620.2001 0.3 0.5 28.5 C42 H26 N F4 429.2 6.921 0.10 42 26 1 4
620.2000 0.4 0.6 17.5 C34 H27 N F9 426.6 4.350 1.29 34 27 1 9
620.1999 0.5 0.8 32.5 C40 H23 N7 F 430.4 8.101 0.03 40 23 7 1
620.1998 0.6 1.0 6.5 C26 H28 N F14 425.2 2.922 5.38 26 28 1 14
620.1997 0.7 1.1 21.5 C32 H24 N7 F6 428.1 5.852 0.29 32 24 7 6
19072019_301_5386
JB-208_19072019_015 142 (3.658) AM2 (Ar,30000.0,556.49,0.00,LS 1)
1: TOF MS ES+
1.35e+007
100
%
0
620.2004
621.2039
620.1368
622.2083
174.1245
248.2004
361.2256
525.1727
619.8981
623.2227
712.1948
809.0287
1016.3021
1052.9625
1215.7756
1355.6322
1520.8357
1570.2012
1892.1362
1990.1305
2195.7607
2287.4463
2423.6980
100 200 300 400 500 600 700 800 900 1000 1100 1200 1300 1400 1500 1600 1700 1800 1900 2000 2100 2200 2300 2400
m/z
F
F
F
F
N
4ad
For Help, press F1
10:55 AM
12/5/2019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8.0715
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7.0930
7.0787
7.0645
7.0164
6.8800
6.8146
6.7996
6.7847
6.7789
6.7635
6.7546
6.7396
6.7245
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6.6468
6.6319
6.5931
6.5786
6.4963
6.4121
2.2907
2.2452
1.9824
1.9631
0.0064
N
4af
1.00
2.08
2.51
4.04
1.80
1.55
1.96
3.57
2.09
1.00
0.96
1.01
6.33
6.29
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
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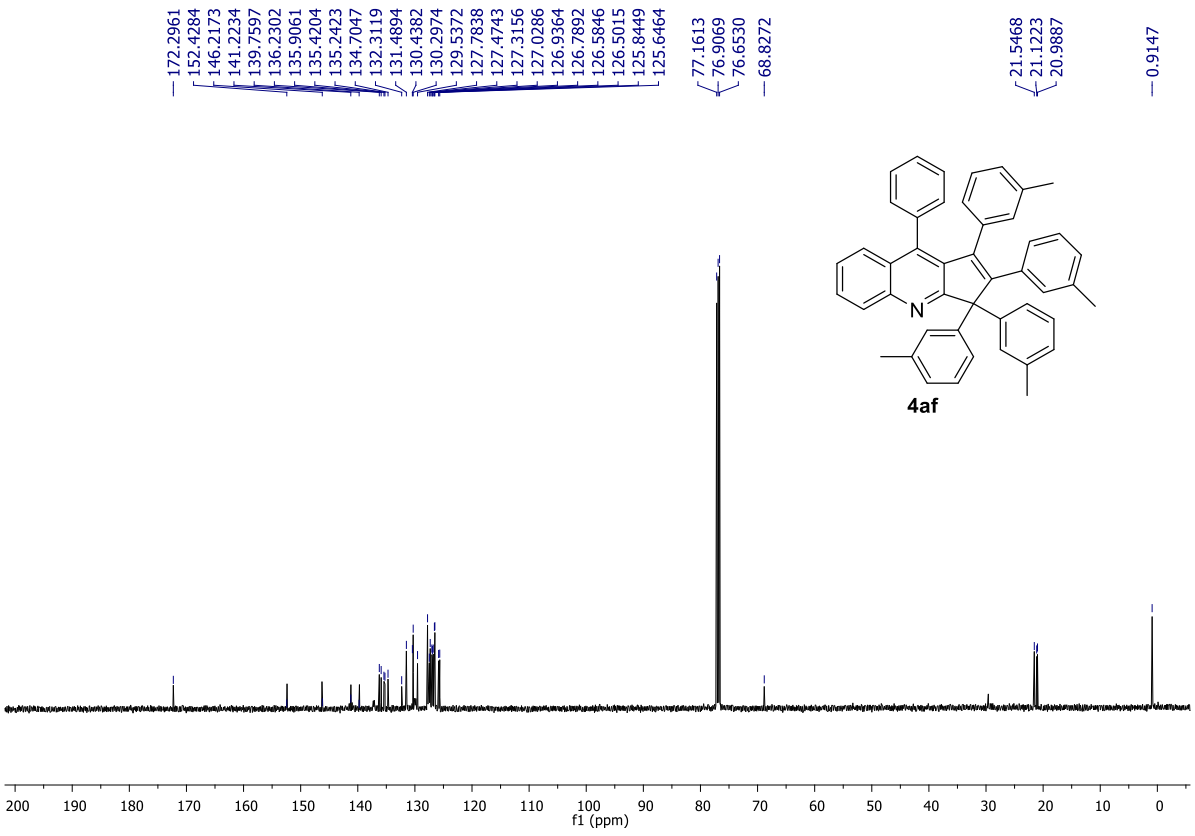
1.0
0.5
0.0
-0.5
f1 (ppm)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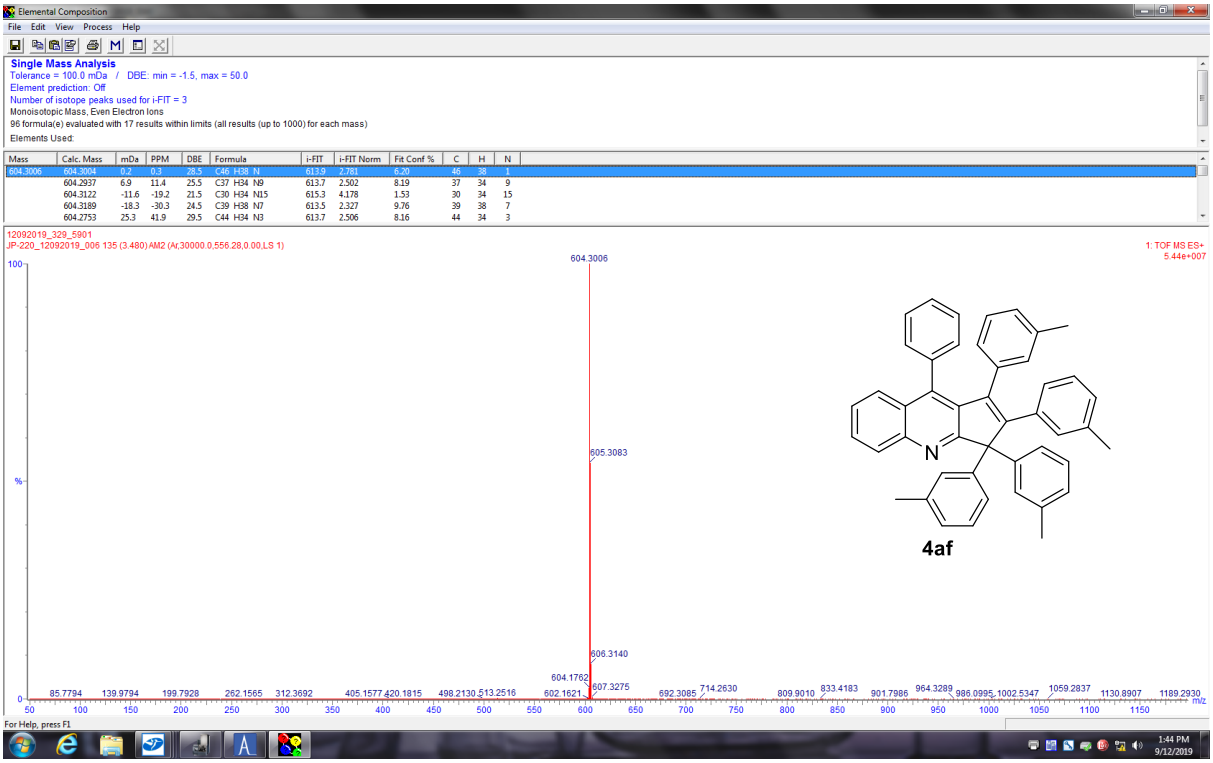
141.2234
139.7597
136.2302
135.9061
135.4204
135.2423
134.7047
132.3119
131.4894
130.4382
130.2974
129.5372
127.7838
127.4743
127.3156
127.0286
126.9364
126.7892
126.5846
126.5015
125.8449
125.6464
77.1613
76.9069
76.6530
68.8272
21.5468
21.1223
20.9887
0.9147
N
4af
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


Elemental Composition
Single Mass Analysis
Tolerance = 100.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3
Monoisotopic Mass, Even Electron Ions
96 formula(e) evaluated with 17 results within limits (all results (up to 1000) for each mass)
Elements Used:
Mass Calc. Mass mDa PPM DBE Formula i-FIT i-FIT Norm Fit Conf % C H N
604.3006 604.3004 0.2 0.3 28.5 C46 H38 N 613.9 2.761 6.20 46 38 1
604.2937 6.9 11.4 25.5 C37 H34 N9 613.7 2.502 8.19 37 34 9
604.3122 -11.6 -19.2 21.5 C30 H34 N15 615.3 4.178 1.53 30 34 15
604.3189 -18.3 -30.3 24.5 C39 H38 N7 613.5 2.327 9.76 39 38 7
604.2753 25.3 41.9 29.5 C44 H34 N3 613.7 2.506 8.16 44 34 3
12092019_329_5901
JP-220_12092019_006 135 (3.480) AM2 (Ar,30000.0,556.28,0.00,LS 1)
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5.44e+007
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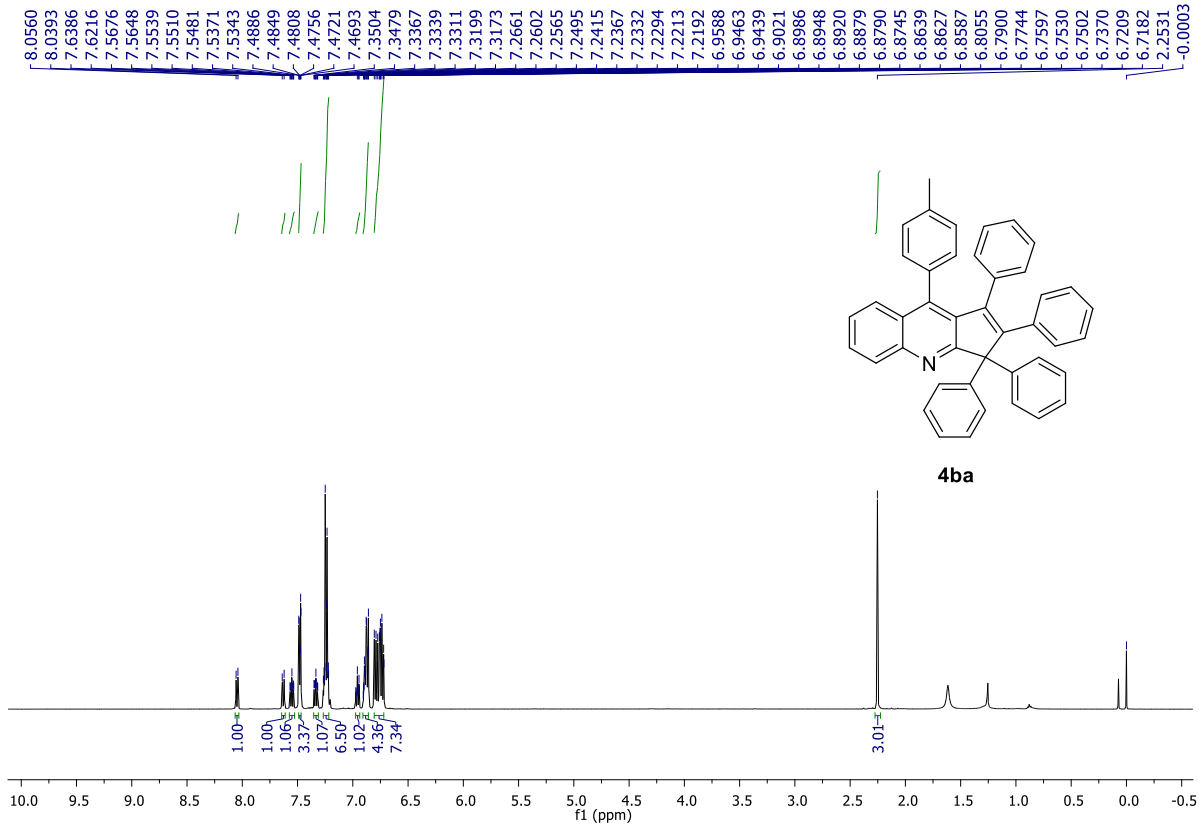
606.3140
604.1762
607.3275
4af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8.0393
7.6386
7.6216
7.5676
7.5648
7.5539
7.5510
7.5481
7.5371
7.5343
7.4886
7.4849
7.4808
7.4756
7.4721
7.4693
7.3504
7.3479
7.3367
7.3339
7.3311
7.3199
7.3173
7.2661
7.2602
7.2565
7.2495
7.2415
7.2367
7.2332
7.2294
7.2213
7.2192
6.9588
6.9463
6.9439
6.9021
6.8986
6.8948
6.8920
6.8879
6.8790
6.8745
6.8639
6.8627
6.8587
6.8055
6.7900
6.7744
6.7597
6.7530
6.7502
6.7370
6.7209
6.7182
2.2531
-0.0003
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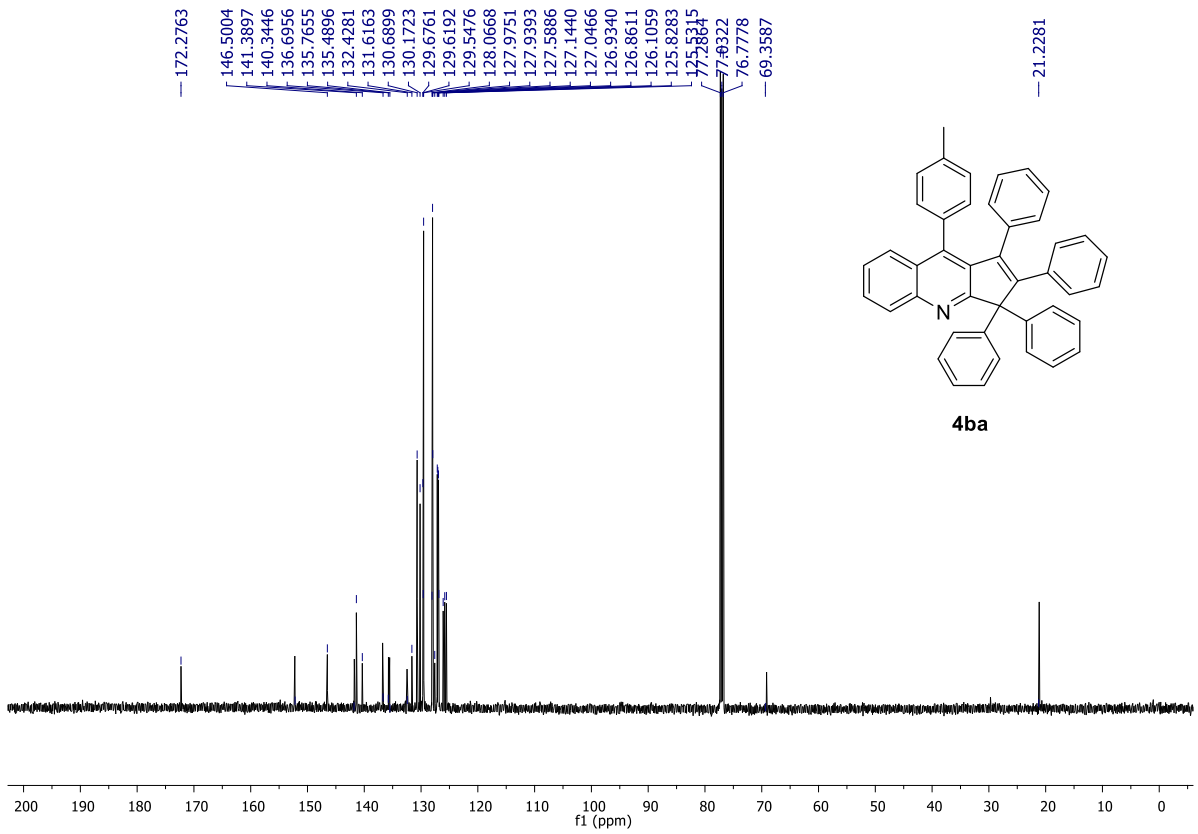
1.00
1.06
3.37
1.07
6.50
1.02
4.36
7.34
3.01
f1 (ppm)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135.4896
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130.1723
129.6761
129.6192
129.5476
128.0668
127.9751
127.9393
127.5886
127.1440
127.0466
126.9340
126.8611
126.1059
125.8283
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77.0322
76.7778
69.3587
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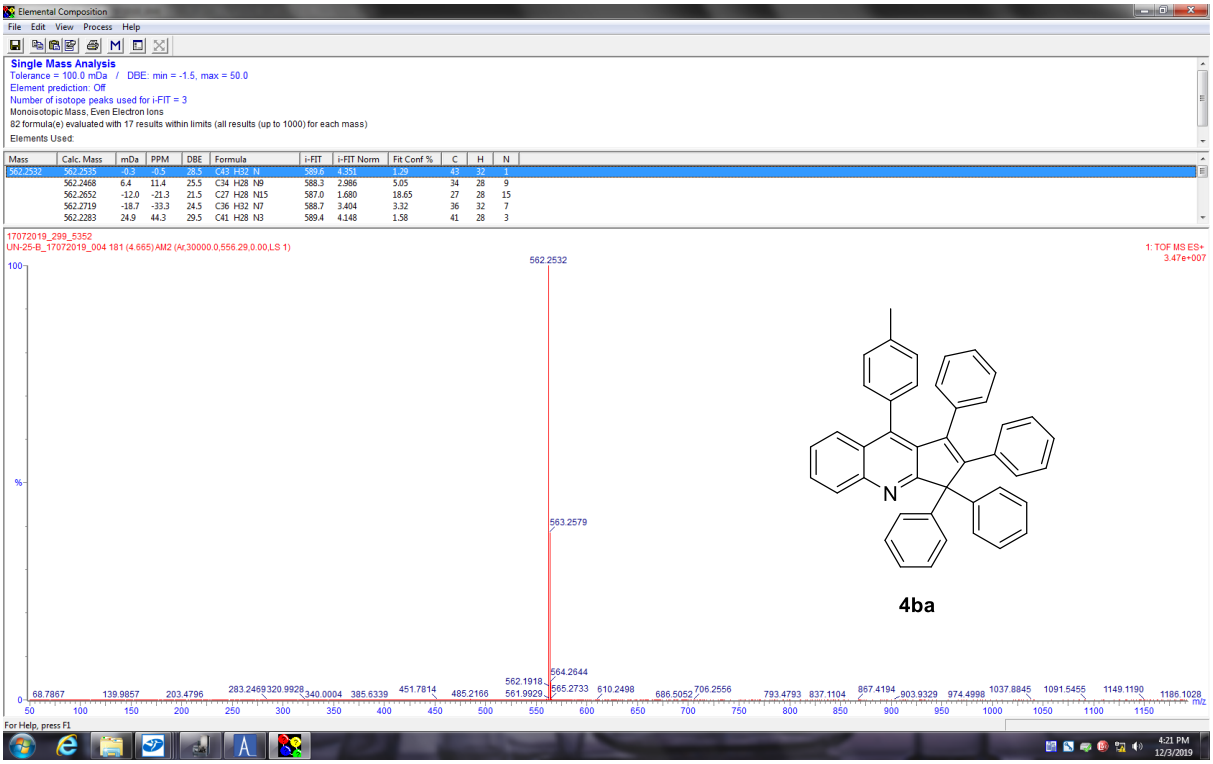
4ba
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


Elemental Composition
File Edit View Process Help
Single Mass Analysis
Tolerance = 100.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3
Monoisotopic Mass, Even Electron Ions
82 formula(e) evaluated with 17 results within limits (all results (up to 1000) for each mass)
Elements Used:
Mass Calc. Mass mDa PPM DBE Formula i-FIT i-FIT Norm Fit Conf % C H N
562.2532 562.2535 -0.3 -0.5 28.5 C43 H32 N 589.6 4.351 1.29 43 32 1
562.2468 6.4 11.4 25.5 C34 H28 N9 588.3 2.986 5.05 34 28 9
562.2652 -12.0 -21.3 21.5 C27 H28 N15 587.0 1.680 18.65 27 28 15
562.2719 -18.7 -33.3 24.5 C36 H32 N7 588.7 3.404 3.32 36 32 7
562.2283 24.9 44.3 29.5 C41 H28 N3 589.4 4.148 1.58 41 28 3
17072019_299_5352
UN-25-B_17072019_004 181 (4.665) AM2 (Ar,30000.0,556.29,0.00,LS 1)
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3.47e+007
100
%
0
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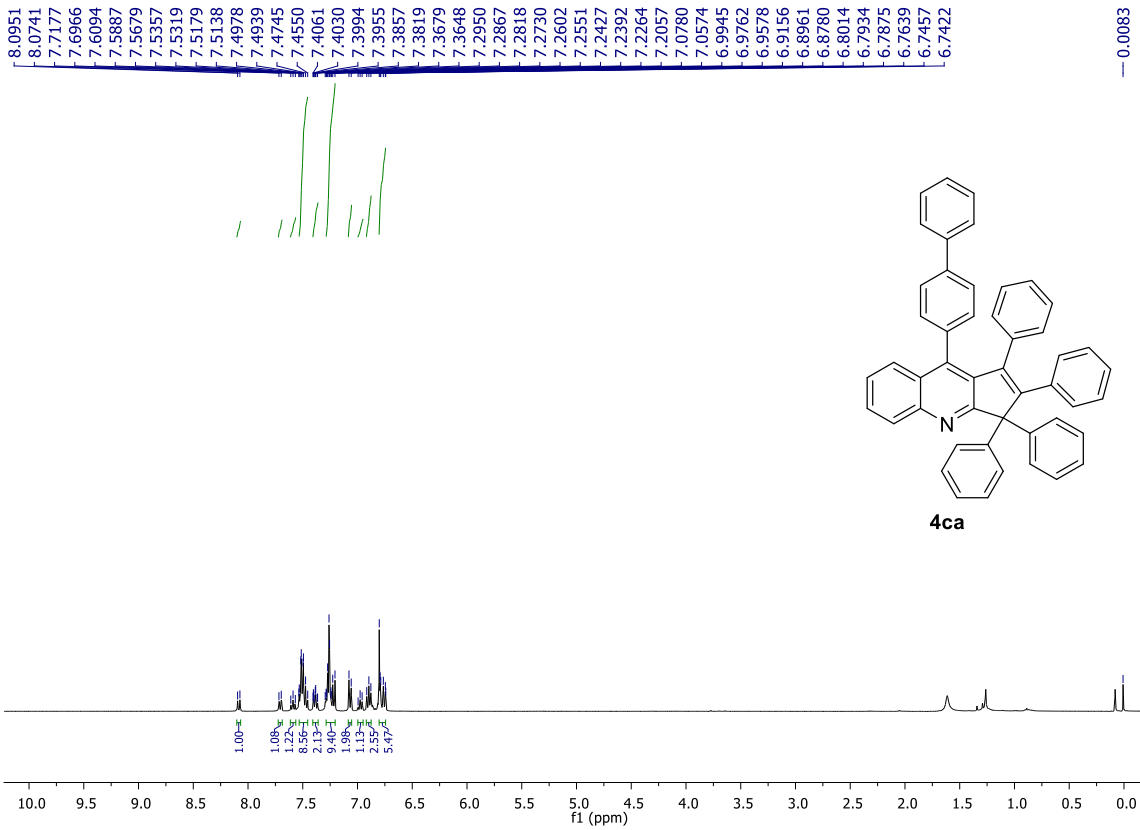
563.2579
564.2644
562.1918
561.9929
565.2733
68.7867
139.9857
203.4796
283.2469
320.9928
340.0004
385.6339
451.7814
485.2166
610.2498
686.5052
706.2556
793.4793
837.1104
867.4194
903.9329
974.4998
1037.8845
1091.5455
1149.1190
1186.1028
m/z
50 100 150 200 250 300 350 400 450 500 550 600 650 700 750 800 850 900 950 1000 1050 1100 1150
N
4ba
For Help, press F1
4:21 PM
12/3/2019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7.6966
7.6094
7.5887
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7.5319
7.5179
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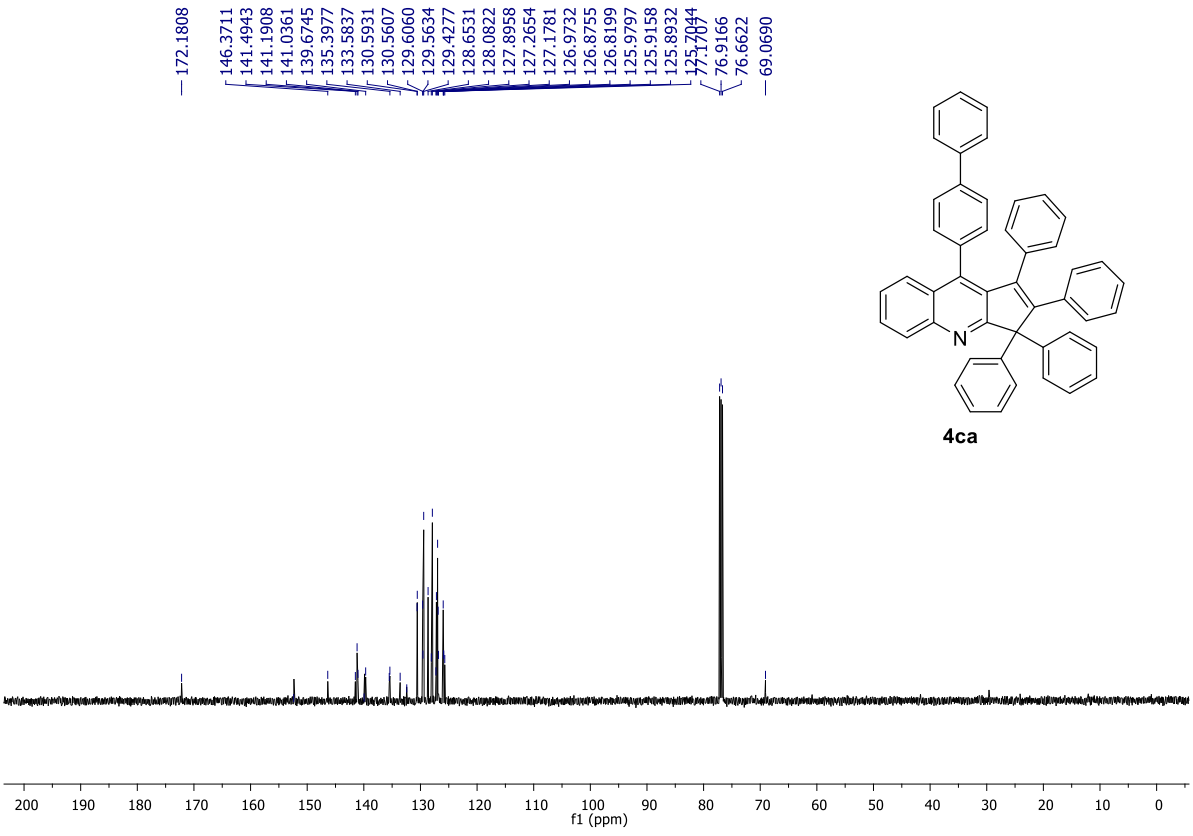
7.4978
7.4939
7.4745
7.4550
7.4061
7.4030
7.3994
7.3955
7.3857
7.3819
7.3679
7.3648
7.2950
7.2867
7.2818
7.2730
7.2602
7.2551
7.2427
7.2392
7.2264
7.2057
7.0780
7.0574
6.9945
6.9762
6.9578
6.9156
6.8961
6.8780
6.8014
6.7934
6.7875
6.7639
6.7457
6.7422
0.0083
N
4ca
1.00
1.08
1.22
8.56
2.13
9.40
1.98
1.13
2.55
5.47
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 -0.5
f1 (ppm)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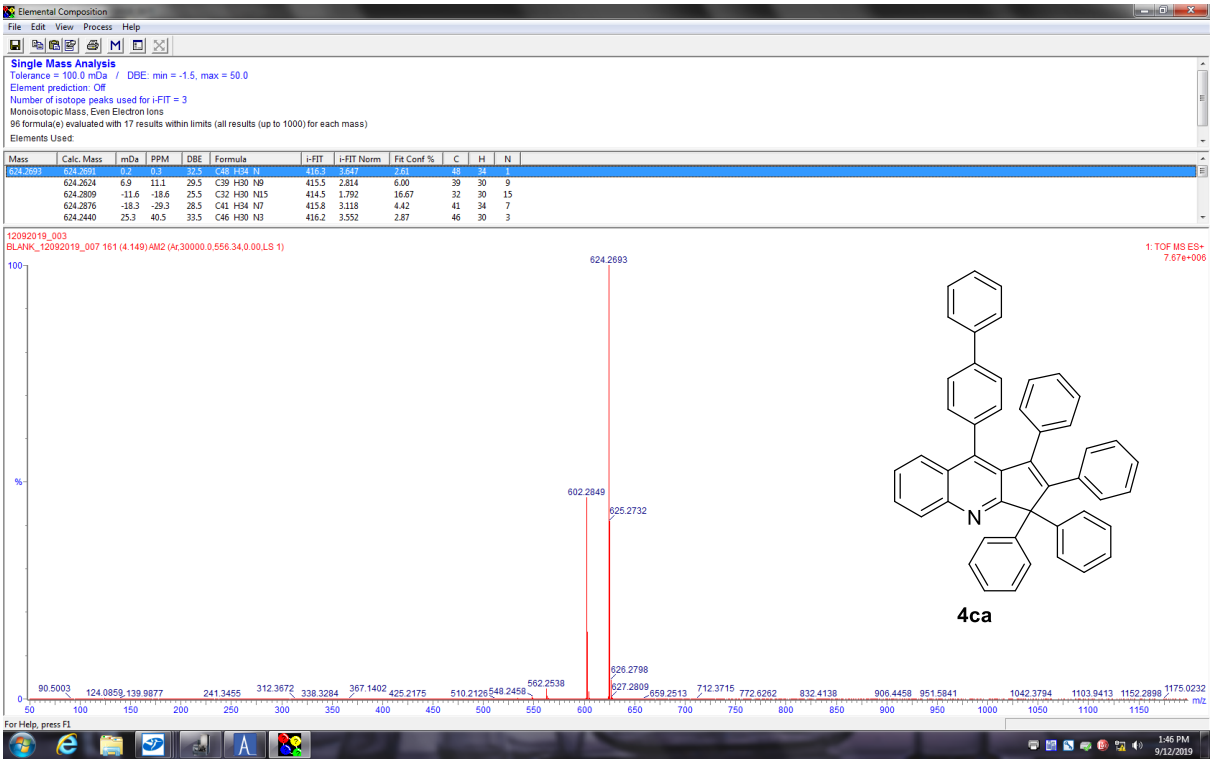
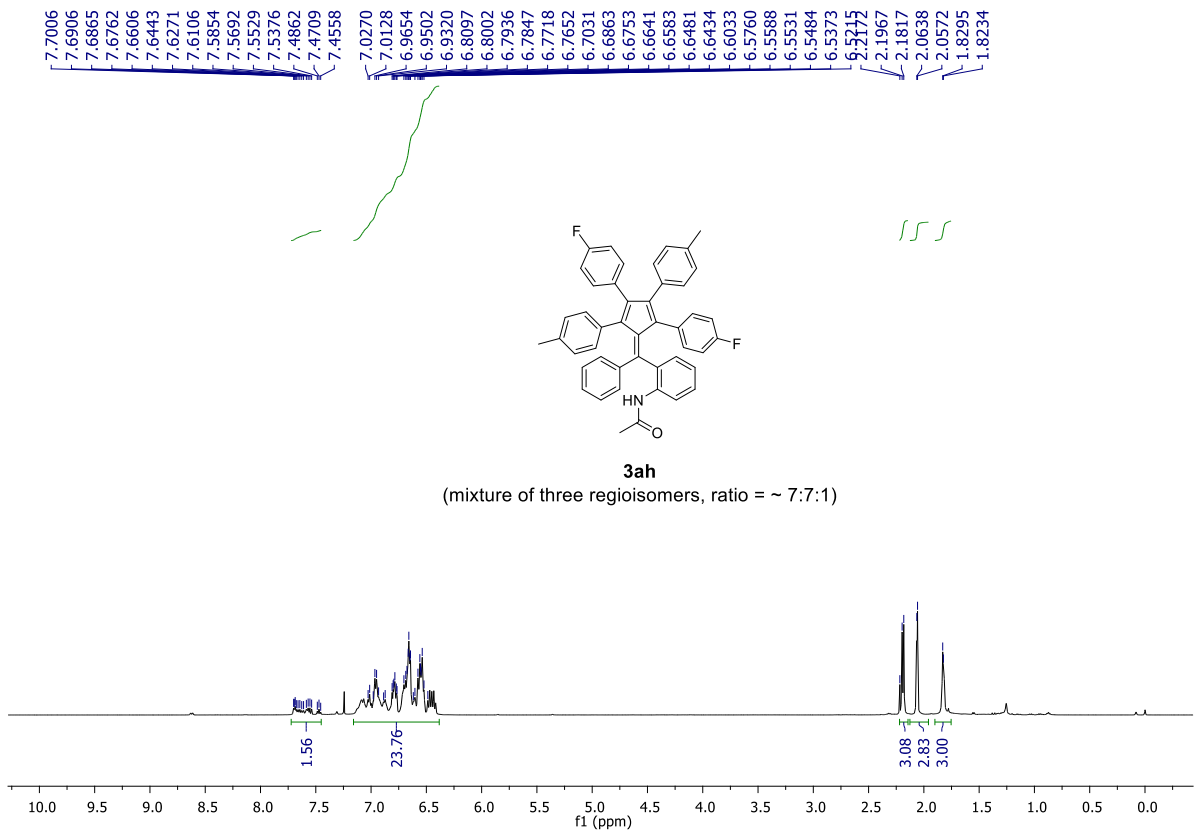
141.1908
141.0361
139.6745
135.3977
133.5837
130.5931
130.5607
129.6060
129.5634
129.4277
128.6531
128.0822
127.8958
127.2654
127.1781
126.9732
126.8755
126.8199
125.9797
125.9158
125.8932
125.7044
77.1707
76.9166
76.6622
69.0690
N
4ca
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

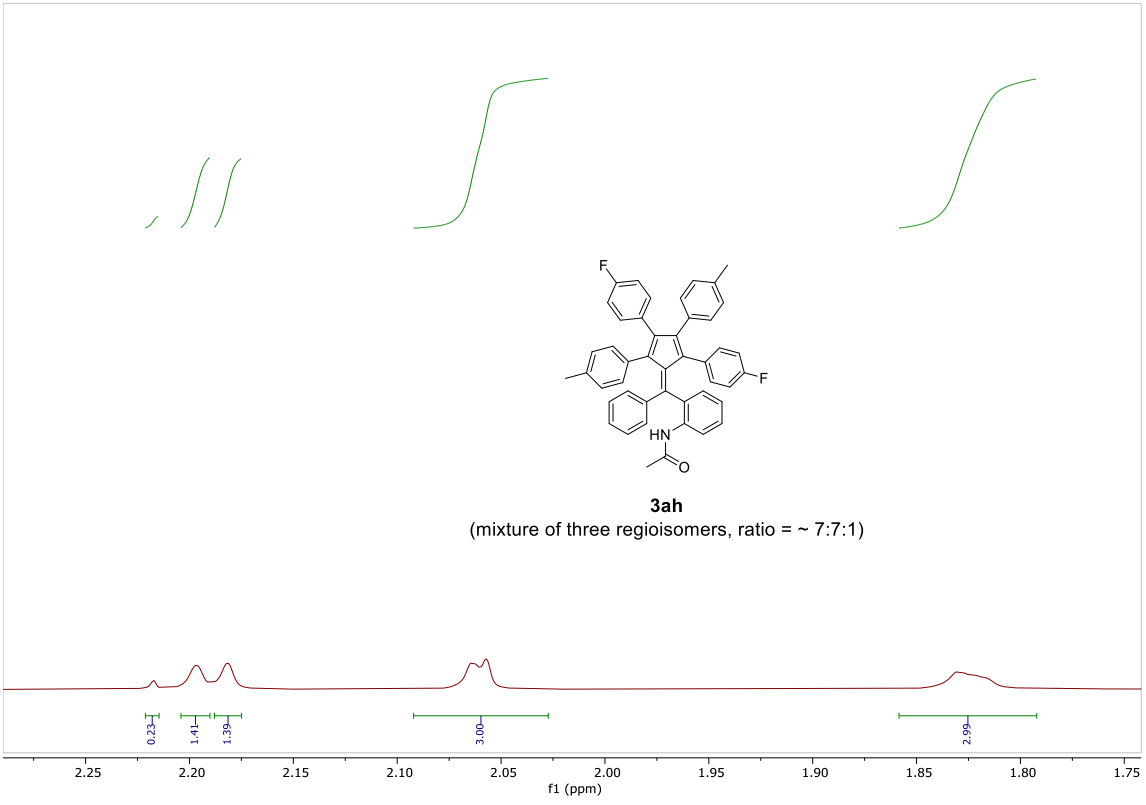

Elemental Composition
File Edit View Process Help
Single Mass Analysis
Tolerance = 100.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3
Monoisotopic Mass, Even Electron Ions
96 formula(e) evaluated with 17 results within limits (all results (up to 1000) for each mass)
Elements Used:
Mass Calc. Mass mDa PPM DBE Formula i-FIT i-FIT Norm Fit Conf % C H N
624.2693 624.2691 0.2 0.3 32.5 C48 H34 N 416.3 3.647 2.61 48 34 1
624.2624 6.9 11.1 29.5 C39 H30 N9 415.5 2.814 6.00 39 30 9
624.2809 -11.6 -18.6 25.5 C32 H30 N15 414.5 1.792 16.67 32 30 15
624.2876 -18.3 -29.3 28.5 C41 H34 N7 415.8 3.118 4.42 41 34 7
624.2440 25.3 40.5 33.5 C46 H30 N3 416.2 3.552 2.87 46 30 3
12092019_003
BLANK_12092019_007 161 (4.149) AM2 (Ar,30000.0,556.34,0.00,LS 1)
1: TOF MS ES+
7.67e+006
624.2693
602.2849
625.2732
626.2798
627.2809
N
4ca
For Help, press F1
1:46 PM
9/12/2019

Determination of ratio of regioisomers:

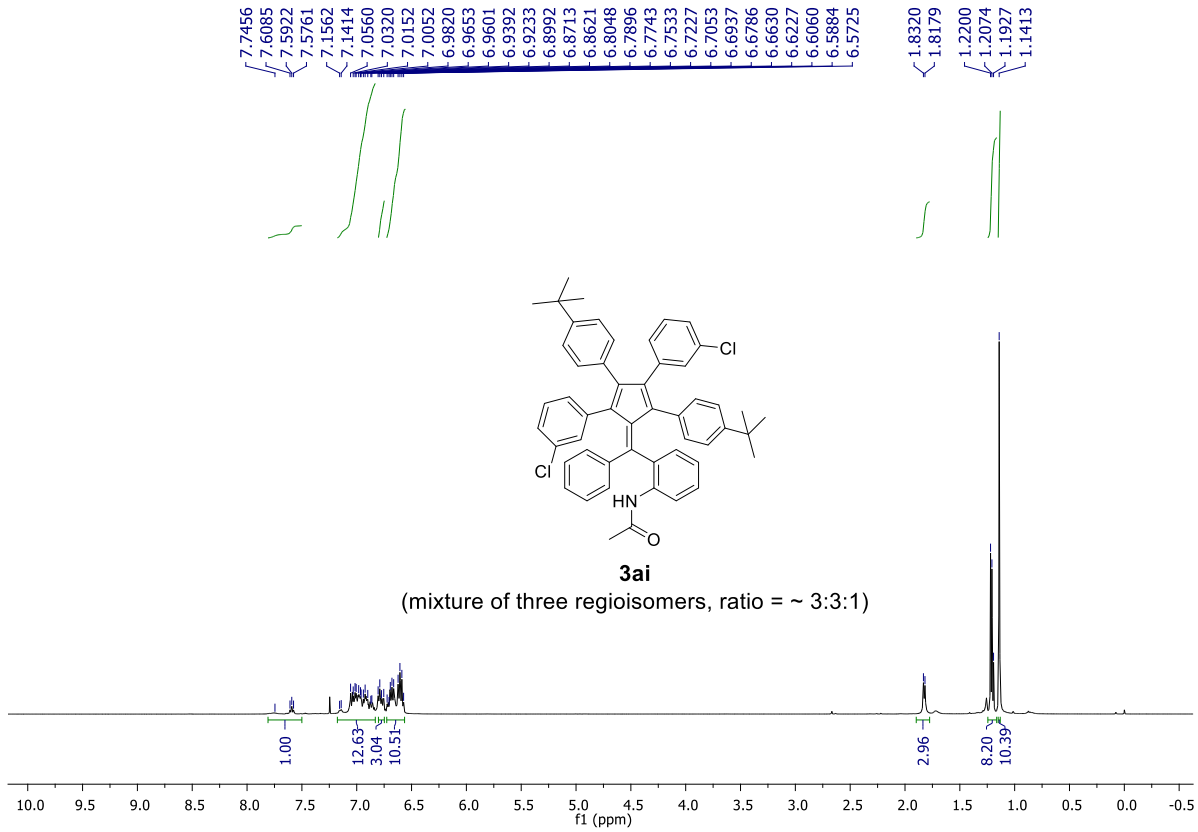


3ah
(mixture of three regioisomers, ratio = ~ 7:7:1)

Expanded form:



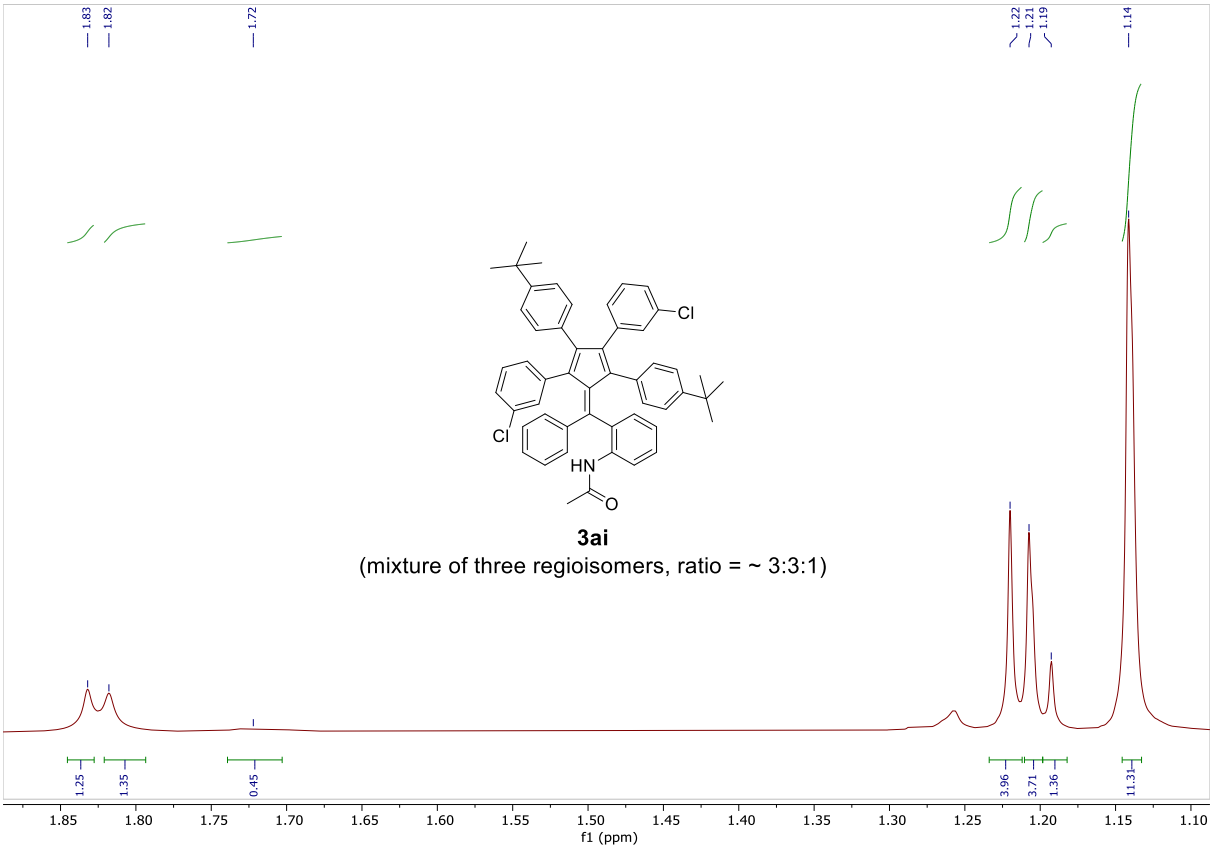
3ah
(mixture of three regioisomers, ratio = ~ 7:7:1)



3ai

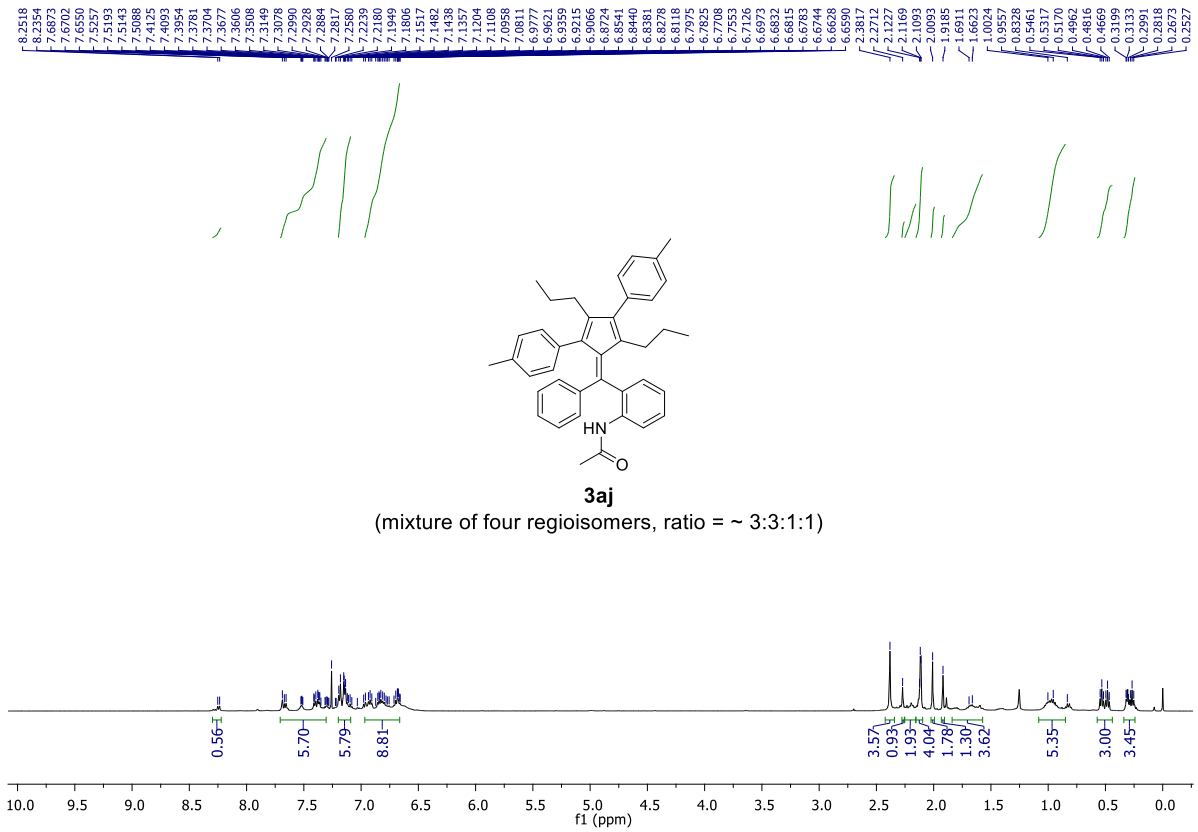
(mixture of three regioisomers, ratio = ~ 3:3:1)

Expanded form:

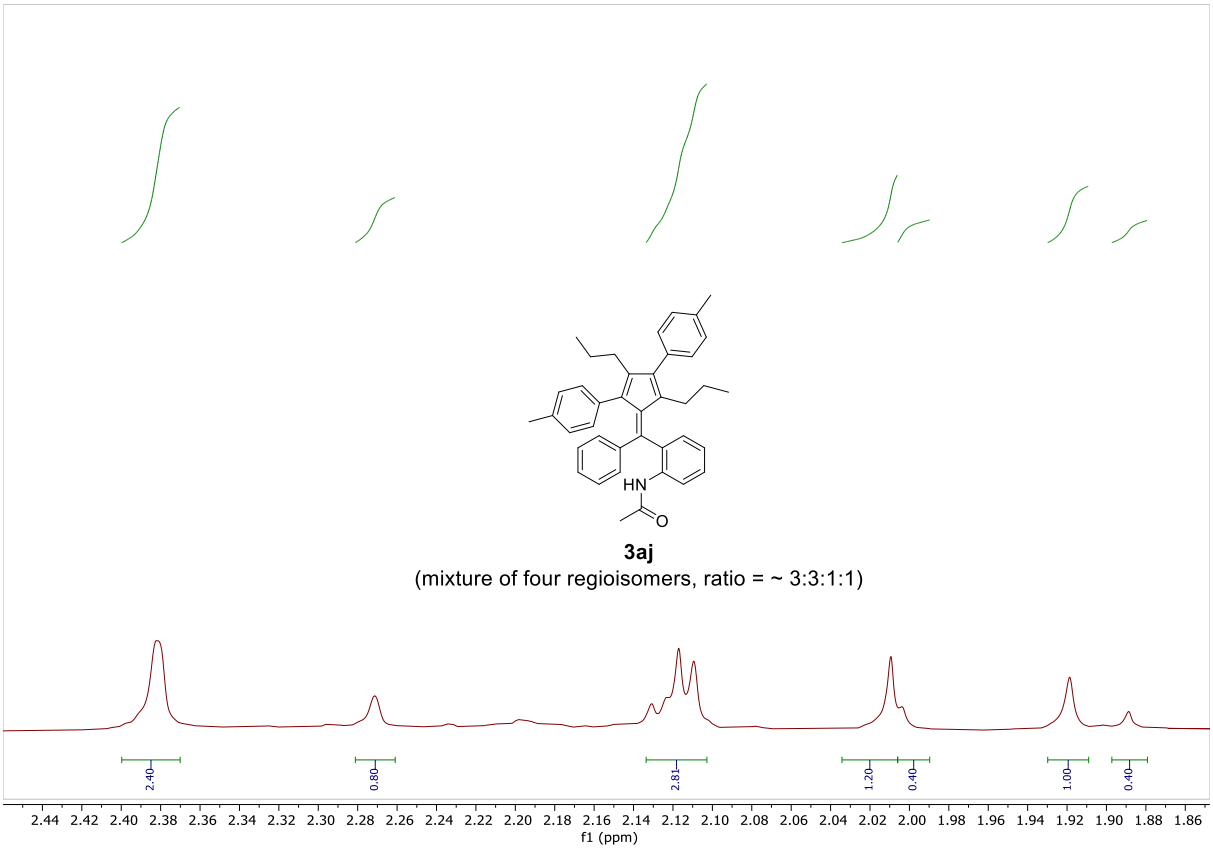


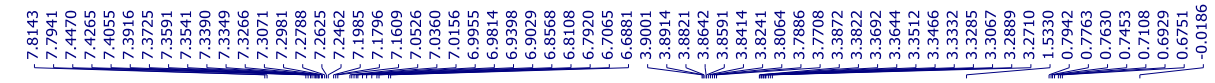
3ai

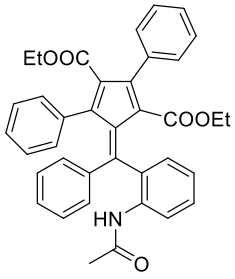
(mixture of three regioisomers, ratio = ~ 3:3:1)



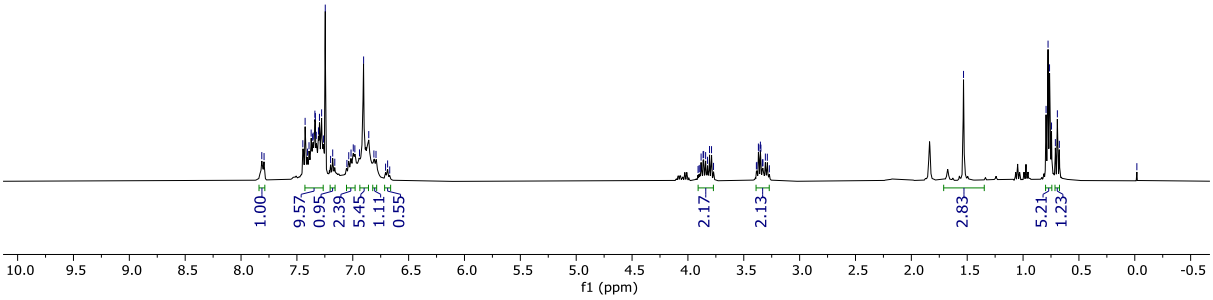
Expanded form:



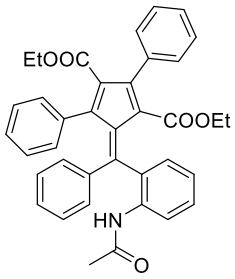




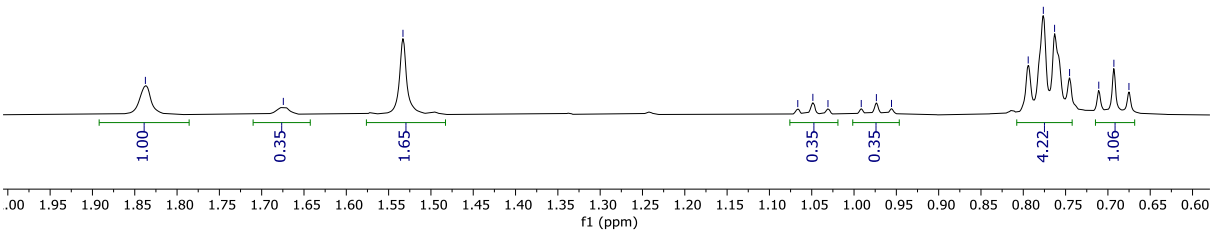
3ak
(mixture of three regioisomers, ratio = ~ 5:3:1)

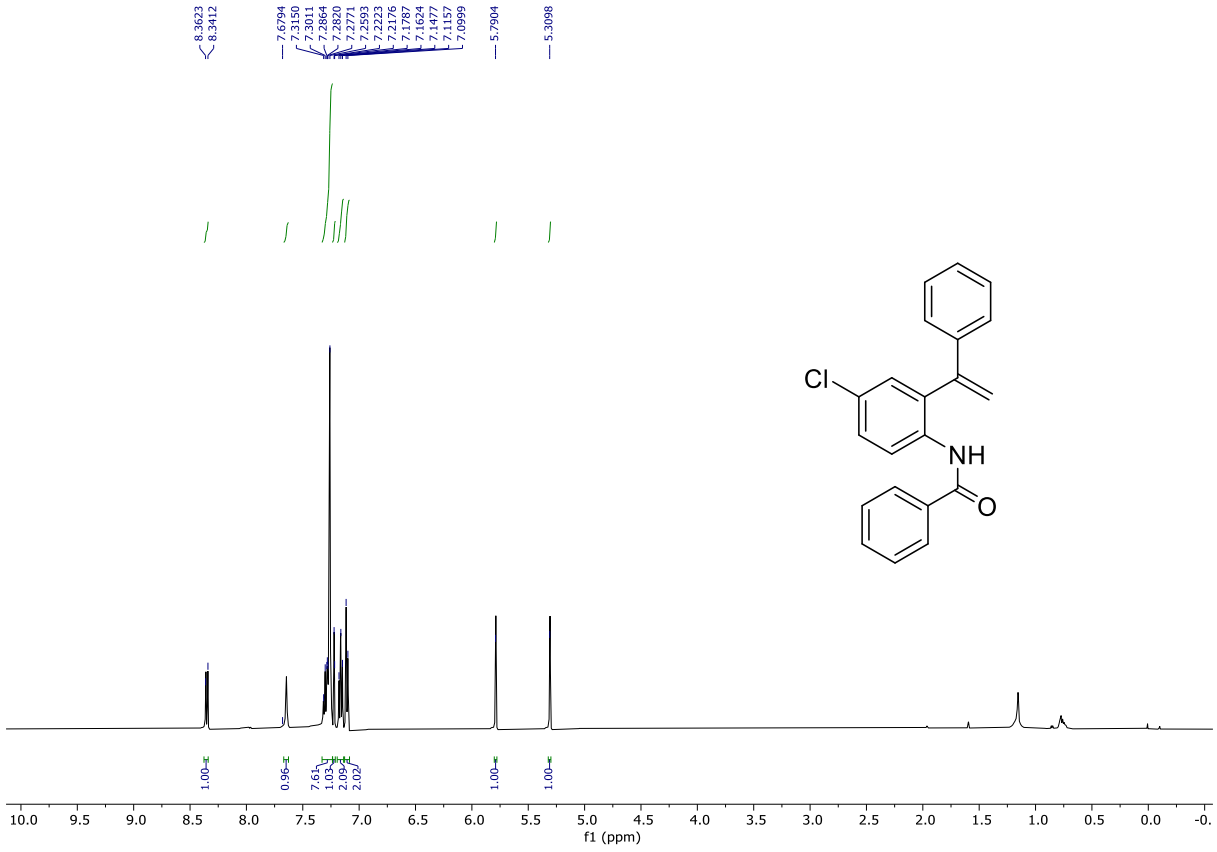


Expanded form:



3ak
(mixture of three regioisomers, ratio = ~ 5:3:1)

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7.3011
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7.2771
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7.2223
7.2176
7.1787
7.1624
7.1477
7.1157
7.0999
5.7904
5.3098
Cl
NH
O
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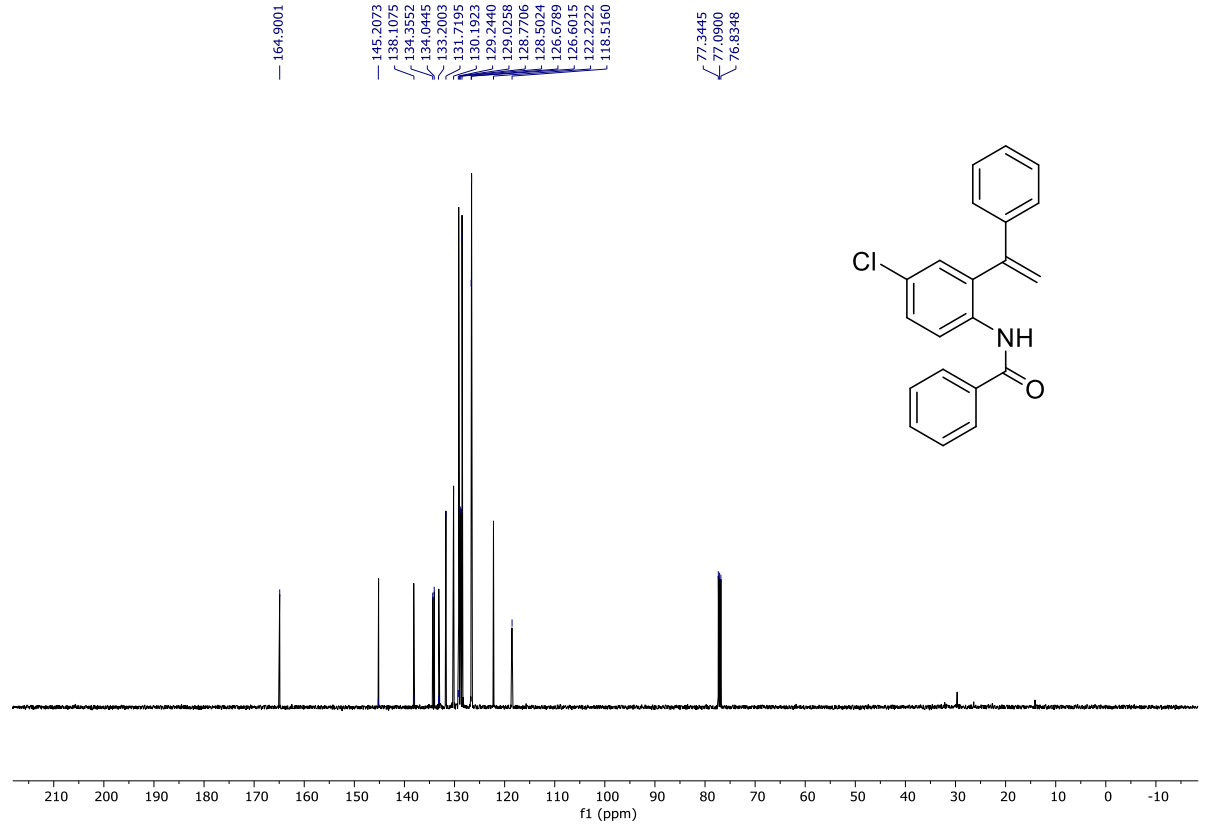
0.96
7.61
1.03
2.09
2.02
1.00
1.00
f1 (ppm)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138.1075
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134.0445
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131.7195
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128.7706
128.5024
126.6789
126.6015
122.2222
118.5160
77.3445
77.0900
76.8348
Cl
NH
O
f1 (ppm)