Ru(II)-Catalyzed Carbonylation Reaction using Ketosulfoxonium Ylide as the Carbonyl Source: Synthesis of indazolo-phthalazinetriones and indazolo-indazolediones

Priya Sonowal, Pratiksha Bhorali, Deep J. Kalita, Supriya Khundrakpam, Bidisha R. Bora and Sanjib Gogoi*

Applied Organic Chemistry, Chemical Sciences & Technology Division, CSIR-North East Institute of Science and Technology, Jorhat-785006, Academy of Scientific and Innovative Research (AcSIR), Ghaziabad-201002, India, Email: skgogoi1@gmail.com, sanjibgogoi@neist.res.in

Supporting Information

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). TLC was performed on Merck TLC Silica gel 60 F_{254} precoated plates. Column chromatography was performed on silica gel (100-200 mesh, Merck). All the heating reactions were performed on oil bath. NMR spectra were recorded on Bruker Avance III 500 or 400 MHz FTNMR spectrometer using tetramethylsilane (TMS) as an internal standard. HRMS data were recorded by mass analyzer of model - Xevo G2-XS Q-TOF, Make-Waters, Software-MassLynx V 4.1. The starting 2-phenyl-2,3-dihydrophthalazine-1,4-diones and 1-phenyl-1,2-dihydro-3*H*-indazol-3-ones were synthesized by following reported procedures.[1]

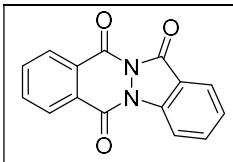
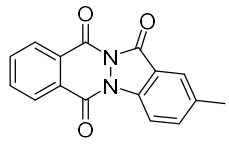
2. Reaction Procedure

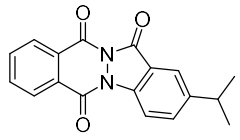
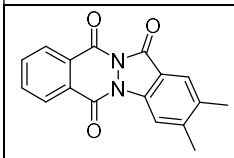
2.1. General procedure for the synthesis of indazolo-phthalazinetriones: A solution of 2-phenyl-2,3-dihydrophthalazine-1,4-diones (0.25 mmol), sulfoxonium ylide (0.25 mmol), $[RuCl_2(p\text{-cymene})]_2$ (5 mol %) and $Cu(OAc)_2.H_2O$ (0.25 mmol) in ^{t}AmOH (4.0 mL) was stirred at 100 °C under open air for 6 hours. The solvent was removed under vacuum and the crude reaction mixture was poured into water and extracted with ethyl acetate (25 mL × 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 vacuum. The crude residue product obtained was purified by silica gel (100-200 mesh) column chromatography using EtOAc/Hexane (1:10) as the eluent.

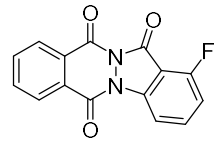
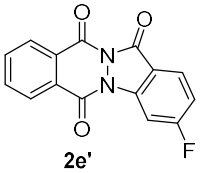
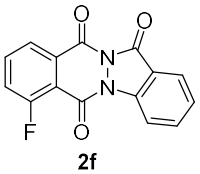
2.2. General procedure for the synthesis of indazolo-indazolediones: A solution of 1-phenyl-1,2-dihydro-3*H*-indazol-3-one (0.25 mmol), sulfoxonium ylide (0.25 mmol), $[RuCl_2(p\text{-cymene})]_2$ (5 mol%) and $Cu(OAc)_2.H_2O$ (0.25 mmol) in ^{t}AmOH (4.0 mL) was stirred at 100 °C under open air for 6 hours. The solvent was removed under vacuum and the crude reaction mixture was poured into water and extracted with ethyl acetate (25 mL × 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

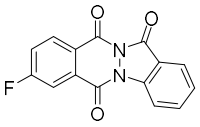
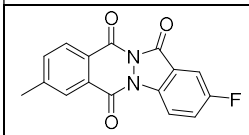
vacuum. The crude residue product obtained was purified by silica gel (100-200 mesh) column chromatography using EtOAc/Hexane (1:10) as the eluent.

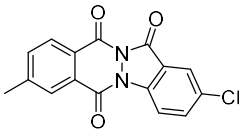
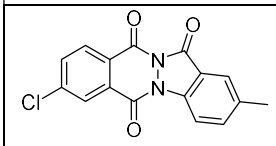
3. Spectral and Analytical Data:

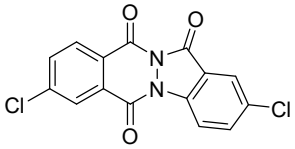
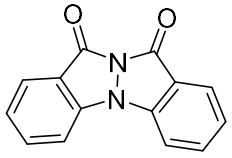
 2a	**13*H*-indazolo[1,2-*b*]phthalazine-6,11,13-trione (2a)[2]:** Following the general procedure **2.1**, the reaction was carried out by heating a mixture of 2-phenyl-2,3-dihydrophthalazine-1,4-dione **1a** (59 mg, 0.25 mmol), sulfoxonium ylide **A** (49 mg, 0.25 mmol), $[RuCl_2(p\text{-cymene})]_2$ (8 mg, 5 mol %) and $Cu(OAc)_2.H_2O$ (50 mg, 0.25 mmol) in ^{t}AmOH (4.0 mL) at 100 ^{o}C for 6 h under air to afford the title compound. The product was purified by flash column chromatography on silica gel (10% EtOAc in hexane); 75% yield (white solid, 49 mg); mp 214-216 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.59 (d, *J* = 8.4 Hz, 1H), 8.52–8.49 (m, 1H), 8.44–8.42 (m, 1H), 8.07 (d, *J* = 7.8 Hz, 1H), 7.97–7.90 (m, 2H), 7.87–7.83 (m, 1H), 7.48 (t, *J* = 7.6 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.8, 154.9, 153.8, 139.2, 136.6, 135.2, 134.7, 129.2, 128.9, 128.8, 128.3, 126.4, 125.4, 116.5, 116.2. HRMS Calcd (ESI) m/z for $C_{15}H_9N_2O_3$: $[M+H]^+$ 265.0613, found: 265.0608.
 2b	**2-methyl-13*H*-indazolo[1,2-*b*]phthalazine-6,11,13-trione (2b)[2]:** Following the general procedure **2.1**, the reaction was carried out by heating a mixture of 2-phenyl-2,3-dihydrophthalazine-1,4-dione **1b** (63 mg, 0.25 mmol), sulfoxonium ylide **A** (49 mg, 0.25 mmol), $[RuCl_2(p\text{-cymene})]_2$ (8 mg, 5 mol %) and $Cu(OAc)_2.H_2O$ (50 mg, 0.25 mmol) in ^{t}AmOH (4.0 mL) at 100 ^{o}C for 6 h under air to afford the title compound. The product was purified by flash column chromatography on silica gel (10% EtOAc in hexane); 73% yield

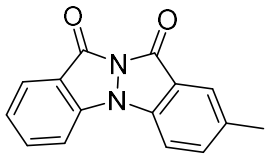
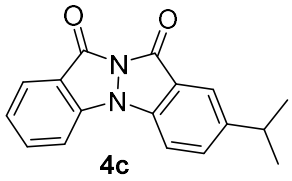
	(pale yellow solid, 51 mg); mp 222-224 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.51–8.41 (m, 3H), 7.96–7.85 (m, 2H), 7.85 (s, 1H), 7.66 (dd, *J* = 8.5, 1.8 Hz, 1H), 2.50 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 157.4, 154.1, 152.7, 137.0, 136.6, 136.0, 134.3, 133.7, 128.4, 128.2, 127.4, 124.1, 115.1, 20.3. HRMS Calcd (ESI) m/z for $C_{16}H_{11}N_2O_3$: $[M+H]^+$ 279.0770, found: 279.0767.
 2c	**2-isopropyl-13*H*-indazolo[1,2-*b*]phthalazine-6,11,13-trione (2c)**[2]: Following the general procedure **2.1**, the reaction was carried out by heating a mixture of 2-phenyl-2,3-dihydrophthalazine-1,4-dione **1c** (70 mg, 0.25 mmol), sulfoxonium ylide **A** (49 mg, 0.25 mmol), $[RuCl_2(p\text{-cymene})]_2$ (8 mg, 5 mol %) and $Cu(OAc)_2.H_2O$ (50 mg, 0.25 mmol) in ^{t}AmOH (4.0 mL) at 100 °C for 6 h under air to afford the title compound. The product was purified by flash column chromatography on silica gel (10% EtOAc in hexane); 74% yield (pale yellow solid, 57 mg); mp 219-221 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.49–8.39 (m, 3H), 7.95–7.87 (m, 3H), 7.70 (dd, *J* = 8.6, 1.8 Hz, 1H), 3.09–3.02 (m, 1H), 1.32 (s, 3H), 1.30 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 158.5, 155.1, 153.7, 148.0, 137.8, 135.9, 135.2, 134.7, 129.3, 129.1, 129.0, 128.4, 122.5, 116.8, 116.2, 34.0, 24.0. HRMS Calcd (ESI) m/z for $C_{18}H_{15}N_2O_3$: $[M+H]^+$ 307.1083, found: 307.1079.
 2d	**2,3-dimethyl-13*H*-indazolo[1,2-*b*]phthalazine-6,11,13-trione (2d)**[2]**:** Following the general procedure **2.1**, the reaction was carried out by heating a mixture of 2-phenyl-2,3-dihydrophthalazine-1,4-diones **1d** (66 mg, 0.25 mmol), sulfoxonium ylide **A** (49 mg, 0.25 mmol), $[RuCl_2(p\text{-cymene})]_2$ (8 mg, 5 mol %) and $Cu(OAc)_2.H_2O$ (50 mg, 0.25 mmol) in ^{t}AmOH (4.0 mL) at 100 °C for 6 h under air to afford the title compound. The product was purified by flash column chromatography on silica gel (10% EtOAc in hexane); 59% yield (white solid, 43 mg); mp 113-115 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.50–8.48 (m, 1H), 8.42–8.36 (m, 1H), 8.36 (s, 1H), 7.94–7.89

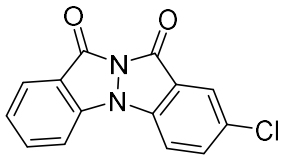
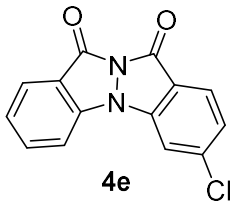
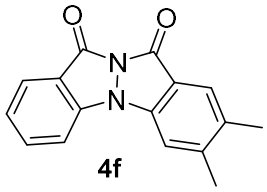
	(m, 2H), 7.78 (s, 1H), 2.47 (s, 3H), 2.38 (s, 3H). HRMS Calcd (ESI) m/z for $C_{17}H_{13}N_2O_3$: $[M+H]^+$ 293.0926, found: 293.0923.
 2e +  2e'	**1-fluoro-13*H*-indazolo[1,2-*b*]phthalazine-6,11,13-trione (2e + 2e′)[2]:** Following the general procedure **2.1**, the reaction was carried out by heating a mixture of 2-phenyl-2,3-dihydrophthalazine-1,4-dione **1e** (64 mg, 0.25 mmol), sulfoxonium ylide **A** (49 mg, 0.25 mmol), $[RuCl_2(p\text{-cymene})]_2$ (8 mg, 5 mol %) and $Cu(OAc)_2.H_2O$ (50 mg, 0.25 mmol) in ^{t}AmOH (4.0 mL) at 100 ^{o}C for 6 h under air to afford the title compound. The product was purified by flash column chromatography on silica gel (10% EtOAc in hexane); 66% yield (white solid, 47 mg); mp 261-263 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.51–8.49 (m, 1H), 8.44–8.39 (m, 1.85H), 8.31–8.28 (m, 0.15H), 8.09–8.05 (m, 0.15H), 7.98–7.92 (m, 2H), 7.84–7.79 (m, 0.85H), 7.13-7.11 (m, 0.15H), 7.11-7.08 (m, 0.85H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 168.8, 166.5, 159.6 (d, J = 263.8 Hz), 154.8, 154.1, 138.6, 135.4, 135.1, 129.5, 128.9, 128.7, 128.6, 115.2 (d, J = 20.0 Hz), 113.0 (J = 18.0 Hz), 112.3 (J = 5.0 Hz), 104.0 (J = 24.0 Hz). HRMS Calcd (ESI) m/z for $C_{15}H_7FN_2O_3$: [M+H]+ 283.0519, found: 283.0515.
 2f	**7-fluoro-13*H*-indazolo[1,2-*b*]phthalazine-6,11,13-trione (2f):** Following the general procedure **2.1**, the reaction was carried out by heating a mixture of 2-phenyl-2,3-dihydrophthalazine-1,4-dione **1f** (64 mg, 0.25 mmol), sulfoxonium ylide **A** (49 mg, 0.25 mmol), $[RuCl_2(p\text{-cymene})]_2$ (8 mg, 5 mol %) and $Cu(OAc)_2.H_2O$ (50 mg, 0.25 mmol) in ^{t}AmOH (4.0 mL) at 100 ^{o}C for 6 h under air to afford the title compound. The product was purified by flash column chromatography on silica gel (10% EtOAc in hexane); 65% yield (pale yellow solid, 46 mg); mp 271-274 ^{o}C. ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.44–8.37 (m, 1H), 8.12 (dd, J = 7.8, 1.2 Hz, 1H), 8.01–7.96 (m, 2H), 7.93–7.89 (m, 1H), 7.83–7.75 (m, 1H), 7.54–7.48 (m, 1H).^{13}C NMR (100 MHz, DMSO-d_6) δ 162.1 (d, J = 271.0

	Hz), 158.2, 153.5, 152.4, 138.8, 137.6, 137.5, 137.2, 131.4, 126.8 (d, J = 22.0 Hz), 125.3, 124.6, 123.5 (d, J = 21.0 Hz), 117.0, 116.1. HRMS Calcd (ESI) m/z for $C_{15}H_8FN_2O_3$: $[M+H]^+$ 283.0519, found: 283.0517.
 2g	**8-fluoro-13*H*-indazolo[1,2-*b*]phthalazine-6,11,13-trione (2g):** Following the general procedure **2.1**, the reaction was carried out by heating a mixture of 2-phenyl-2,3-dihydrophthalazine-1,4-dione **1g** (64 mg, 0.25 mmol), sulfoxonium ylide **A** (49 mg, 0.25 mmol), $[RuCl_2(p\text{-cymene})]_2$ (8 mg, 5 mol %) and $Cu(OAc)_2.H_2O$ (50 mg, 0.25 mmol) in ^{t}AmOH (4.0 mL) at 100 ^{o}C for 6 h under air to afford the title compound. The product was purified by flash column chromatography on silica gel (10% EtOAc in hexane); 68% yield (pale yellow solid, 48 mg); mp 278-280 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.60–8.53 (m, 2H), 8.10–8.07 (m, 2H), 7.89–7.85 (m, 1H), 7.62–7.49 (m, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 166.7 (d, J = 253.0 Hz), 158.1, 154.1, 152.9, 139.2, 136.9, 132.7 (d, J = 9.4 Hz), 131.9, 126.9, 126.7, 125.7, 122.7 (d, J = 23.0 Hz), 116.8, 116.4, 115.2 (d, J = 23.0 Hz). HRMS Calcd (ESI) m/z for $C_{15}H_8FN_2O_3$: $[M+H]^+$ 283.0519, found: 283.0516.
 2h	**2-fluoro-8-methyl-13*H*-indazolo[1,2-*b*]phthalazine-6,11,13-trione (2h):** Following the general procedure **2.1**, the reaction was carried out by heating a mixture of 2-phenyl-2,3-dihydrophthalazine-1,4-dione **1h** (67 mg, 0.25 mmol), sulfoxonium ylide **A** (49 mg, 0.25 mmol), $[RuCl_2(p\text{-cymene})]_2$ (8 mg, 5 mol %) and $Cu(OAc)_2.H_2O$ (50 mg, 0.25 mmol) in ^{t}AmOH (4.0 mL) at 100 ^{o}C for 6 h under air to afford the title compound. The product was purified by flash column chromatography on silica gel (10% EtOAc in hexane); 70% yield (white solid, 51 mg); mp 221-224 ^{o}C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.59–8.57 (m, 1H), 8.37–8.19 (m, 2H), 7.74–7.70 (m, 2H), 7.55 (t, J = 9.3 Hz, 1H), 2.60 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 160.6 (J = 247.5 Hz), 157.3, 154.9, 153.9, 147.1, 146.5,

	136.5, 135.9, 129.5, 128.7, 126.3, 124.6 (d, J = 24.6 Hz), 124.4 (d, J = 24.6 Hz), 118.2 (d, J = 8.8 Hz), 111.2 (d, J = 23.8 Hz), 22.1. HRMS Calcd (ESI) m/z for $C_{16}H_{10}FN_2O_3$: $[M+H]^+$ 297.0675, found: 297.0673.
 **2i**	**2-chloro-8-methyl-13*H*-indazolo[1,2-*b*]phthalazine-6,11,13-trione (2i):** Following the general procedure **2.1**, the reaction was carried out by heating a mixture of 2-phenyl-2,3-dihydrophthalazine-1,4-dione **1i** (72 mg, 0.25 mmol), sulfoxonium ylide **A** (49 mg, 0.25 mmol), $[RuCl_2(p\text{-cymene})]_2$ (8 mg, 5 mol %) and $Cu(OAc)_2.H_2O$ (50 mg, 0.25 mmol) in ^{t}AmOH (4.0 mL) at 100 ^{o}C for 6 h under air to afford the title compound. The product was purified by flash column chromatography on silica gel (10% EtOAc in hexane); 71% yield (pale yellow solid, 55 mg); mp 197-200 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.53 (dd, J = 8.8, 2.5 Hz, 1H), 8.38–8.19 (m, 2H), 8.02 (d, J = 2.0 Hz, 1H), 7.79–7.71 (m, 2H), 2.60 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.0, 154.9, 154.1, 147.2, 146.7, 137.6, 136.8, 136.0, 132.4, 129.6, 128.7, 126.3, 125.0, 118.3, 117.7, 22.2. HRMS Calcd (ESI) m/z for $C_{16}H_{10}ClN_2O_3$: $[M+H]^+$ 313.0380, found: 313.0376.
 **2j**	**8-chloro-2-methyl-13*H*-indazolo[1,2-*b*]phthalazine-6,11,13-trione (2j):** Following the general procedure **2.1**, the reaction was carried out by heating a mixture of 2-phenyl-2,3-dihydrophthalazine-1,4-diones **1j** (72 mg, 0.25 mmol), sulfoxonium ylide **A** (49 mg, 0.25 mmol), $[RuCl_2(p\text{-cymene})]_2$ (8 mg, 5 mol %) and $Cu(OAc)_2.H_2O$ (50 mg, 0.25 mmol) in ^{t}AmOH (4.0 mL) at 100 ^{o}C for 6 h under air to afford the title compound. The product was purified by flash column chromatography on silica gel (10% EtOAc in hexane); 62% yield (pale yellow solid, 48 mg); mp 226-228 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.46–8.43 (m, 2H), 8.39 (d, J = 2.0 Hz, 1H), 7.87–7.84 (m, 2H), 7.68–6.66 (m, 1H), 2.51 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.1, 158.1, 142.4, 138.0, 135.4, 130.9, 128.3,

	125.7, 116.1, 21.3. HRMS Calcd (ESI) m/z for $C_{16}H_{10}ClN_2O_3$: $[M+H]^+$ 313.0380, found: 313.0377.
 **2k**	**2,8-dichloro-13*H*-indazolo[1,2-*b*]phthalazine-6,11,13-trione (2k):** Following the general procedure **2.1**, the reaction was carried out by heating a mixture of 2-phenyl-2,3-dihydrophthalazine-1,4-dione **1k** (76 mg, 0.25 mmol), sulfoxonium ylide **A** (49 mg, 0.25 mmol), $[RuCl_2(p\text{-cymene})]_2$ (8 mg, 5 mol%) and $Cu(OAc)_2.H_2O$ (50 mg, 0.25 mmol) in ^{t}AmOH (4.0 mL) at 100 ^{o}C for 6 h under air to afford the title compound. The product was purified by flash column chromatography on silica gel (10% EtOAc in hexane); 67% yield (pale yellow solid, 56 mg); mp 231-233 ^{o}C. ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.41 (dd, *J* = 8.8, 1.8 Hz, 1H), 8.27 (dd, *J* = 8.4, 5.4 Hz, 1H), 8.21 (d, *J* = 3.9 Hz, 1H), 8.06–8.01 (m, 2H), 7.96–7.93 (m, 1H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 156.9, 154.6, 153.3, 141.0, 140.5, 137.7, 137.2, 137.1, 135.7, 135.3, 128.0, 127.6, 124.8, 118.6, 117.9. HRMS Calcd (ESI) m/z for $C_{15}H_7Cl_2N_2O_3$: $[M+H]^+$ 313.0380, found: 313.0377.
 **4a**	**10*H*,12*H*-indazolo[1,2-*a*]indazole-10,12-dione (4a)[3]:** Following the general procedure **2.2**, the reaction was carried out by heating a mixture of **3a** (52 mg, 0.25 mmol), sulfoxonium ylide **A** (49 mg, 0.25 mmol), $[RuCl_2(p\text{-cymene})]_2$ (8 mg, 5 mol%) and $Cu(OAc)_2.H_2O$ (50 mg, 0.25 mmol) in ^{t}AmOH (4.0 mL) at 100 ^{o}C for 6 h under air to afford the title compound. The product was purified by flash column chromatography on silica gel (10% EtOAc in hexane); 75% yield (pale yellow solid, 44 mg); mp 192-195 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98 (d, *J* = 7.4 Hz, 2H), 7.78–7.75 (m, 2H), 7.51 (d, *J* = 8.2 Hz, 2H), 7.30–7.27 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.3, 141.7, 135.9, 126.8, 123.7, 120.8, 110.5. HRMS Calcd (ESI) m/z for $C_{14}H_9N_2O_2$: $[M+H]^+$ 237.0664, found: 237.0668.

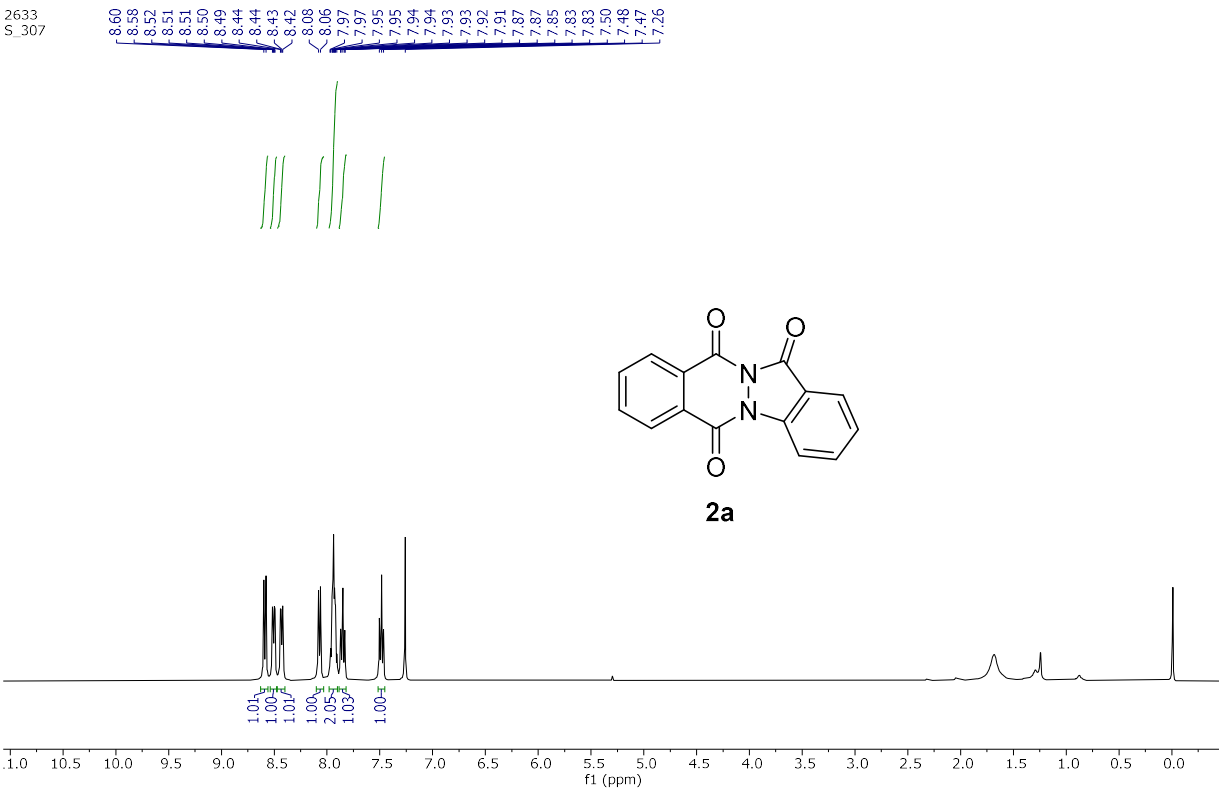
 **4b**	**2-methyl-10*H*,12*H*-indazolo[1,2-*a*]indazole-10,12-dione (4b):** Following the general procedure **2.2**, the reaction was carried out by heating a mixture of **3b** (56 mg, 0.25 mmol), sulfoxonium ylide **A** (49 mg, 0.25 mmol), $[RuCl_2(p\text{-cymene})]_2$ (8 mg, 5 mol%) and $Cu(OAc)_2.H_2O$ (50 mg, 0.25 mmol) in ^{t}AmOH (4.0 mL) at 100 ^{o}C for 6 h under air to afford the title compound. The product was purified by flash column chromatography on silica gel (10% EtOAc in hexane); 72% yield (pale yellow solid, 45 mg); mp 214-216 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93 (d, *J* = 7.9 Hz, 1H), 7.76–7.69 (m, 2H), 7.55 (dd, *J* = 8.5, 1.7 Hz, 1H), 7.45 (d, *J* = 8.3 Hz, 1H), 7.38 (d, *J* = 8.4 Hz, 1H), 7.26–7.22 (m, 1H), 2.42 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.6, 142.1, 140.3, 137.3, 136.0, 134.0, 126.8, 126.5, 123.6, 121.0, 120.8, 110.6, 110.5, 21.1. HRMS Calcd (ESI) m/z for $C_{15}H_{11}N_2O_2$: $[M+H]^+$ 251.0821, found: 251.0817.
 **4c**	**2-isopropyl-10*H*,12*H*-indazolo[1,2-*a*]indazole-10,12-dione (4c):** Following the general procedure **2.2**, the reaction was carried out by heating a mixture of **3c** (63 mg, 0.25 mmol), sulfoxonium ylide **A** (49 mg, 0.25 mmol), $[RuCl_2(p\text{-cymene})]_2$ (8 mg, 5 mol%) and $Cu(OAc)_2.H_2O$ (50 mg, 0.25 mmol) in ^{t}AmOH (4.0 mL) at 100 ^{o}C for 6 h under air to afford the title compound. The product was purified by flash column chromatography on silica gel (10% EtOAc in hexane); 68% yield (pale yellow solid, 47 mg); mp 241-244 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96–7.94 (m, 1H), 7.83 (d, *J* = 1.8 Hz, 1H), 7.75–7.71 (m, 1H), 7.61 (dd, *J* = 8.5, 1.8 Hz, 1H), 7.48–7.40 (m, 2H), 7.25–7.22 (m, 1H), 2.99 (septet, *J* = 8.0 Hz, 1H), 1.27 (d, *J* = 8.0 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.8, 157.7, 145.3, 142.2, 140.6, 136.0, 135.3, 126.9, 123.9, 123.7, 121.1, 120.9, 110.7, 33.7, 24.1.

 4d	**2-chloro-10*H*,12*H*-indazolo[1,2-*a*]indazole-10,12-dione (4d):** Following the general procedure **2.2**, the reaction was carried out by heating a mixture of **3d** (61 mg, 0.25 mmol), sulfoxonium ylide **A** (49 mg, 0.25 mmol), $[RuCl_2(p\text{-cymene})]_2$ (8 mg, 5 mol%) and $Cu(OAc)_2.H_2O$ (50 mg, 0.25 mmol) in ^{t}AmOH (4.0 mL) at 100 ^{o}C for 6 h under air to afford the title compound. The product was purified by flash column chromatography on silica gel (10% EtOAc in hexane); 62% yield (pale yellow solid, 42 mg); mp 225-228 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03–7.96 (m, 2H), 7.80–7.70 (m, 2H), 7.47 (t, *J* = 8.6 Hz, 2H), 7.31–7.30 (d, *J* = 4.6 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.3, 152.1, 146.2, 136.3, 127.2, 126.6, 124.2, 111.8, 110.6. HRMS Calcd (ESI) m/z for $C_{14}H_8ClN_2O_2$: $[M+H]^+$ 271.0274, found: 271.0276.
 4e	**1-chloro-10*H*,12*H*-indazolo[1,2-*a*]indazole-10,12-dione (4e):** Following the general procedure **2.2**, the reaction was carried out by heating a mixture of **3e** (61 mg, 0.25 mmol), sulfoxonium ylide **A** (49 mg, 0.25 mmol), $[RuCl_2(p\text{-cymene})]_2$ (8 mg, 5 mol%) and $Cu(OAc)_2.H_2O$ (50 mg, 0.25 mmol) in ^{t}AmOH (4.0 mL) at 100 ^{o}C for 6 h under air to afford the title compound. The product was purified by flash column chromatography on silica gel (10% EtOAc in hexane); 54% yield (pale yellow solid, 37 mg); mp 232-234 ^{o}C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.00 (d, *J* = 8.0 Hz, 1H), 7.92 (d, *J* = 8.0 Hz, 1H), 7.80 (t, *J* = 7.0 Hz, 1H), 7.51 (s, 1H), 7.50 (d, *J* = 8.5 Hz, 1H), 7.33 (t, *J* = 7.5 Hz, 1H), 7.22-7.26 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.1, 156.5, 141.5, 136.2, 135.5, 131.4, 130.1, 128.6, 127.9, 127.1, 124.5, 124.3, 110.9, 110.7. HRMS Calcd (ESI) m/z for $C_{14}H_8ClN_2O_2$: $[M+H]^+$ 271.0274, found: 271.0272.
 4f	**1,2-dimethyl-10*H*,12*H*-indazolo[1,2-*a*]indazole-10,12-dione (4f):** Following the general procedure **2.2**, the reaction was carried out by heating a mixture of **3f** (56 mg, 0.25 mmol), sulfoxonium ylide **A** (49 mg, 0.25 mmol), $[RuCl_2(p\text{-cymene})]_2$ (8 mg, 5 mol%) and

	$Cu(OAc)_2.H_2O$ (50 mg, 0.25 mmol) in ^tAmOH (4.0 mL) at 100 ^oC for 6 h under air to afford the title compound. The product was purified by flash column chromatography on silica gel (10% EtOAc in hexane); 60% yield (pale yellow solid, 40 mg); mp 183-186 ^oC. ^1H NMR (400 MHz, $CDCl_3$) δ 7.95 (d, *J* = 7.8 Hz, 1H), 7.75–7.66 (m, 2H), 7.48 (d, *J* = 8.2 Hz, 1H), 7.24–7.21 (m, 2H), 2.44 (s, 3H), 2.32 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.3, 157.8, 147.0, 142.1, 135.9, 133.3, 126.8, 126.6, 123.5, 111.3, 110.7, 21.6, 19.8. HRMS Calcd (ESI) m/z for $C_{16}H_{13}N_2O_2$: $[M+H]^+$ 265.0977, found: 265.0971.

References

1. (a) R. Prakash, B. R. Bora, R. C. Boruah and S. Gogoi, *Org. Lett.* **2018**, *20*, 2297−2300; (b) K. Gogoi, B. R. Bora, G. Borah, B. Sarma and S. Gogoi, *Chem. Commun.* **2021**, *57*, 1388−1391.
2. P. Karishma, A. Gogia, S. K. Mandal and R. Sakhuja, *Adv. Synth. Catal.* **2021**, *363*, 762–775.
3. Z. Wang, B. Yu, Y. Cui, X. Sun and W. Bao, *Chin. J. Chem.* **2011**, *29*, 2769–2774.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O
N
N
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1.00
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1.00
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1.03
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f1 (ppm)

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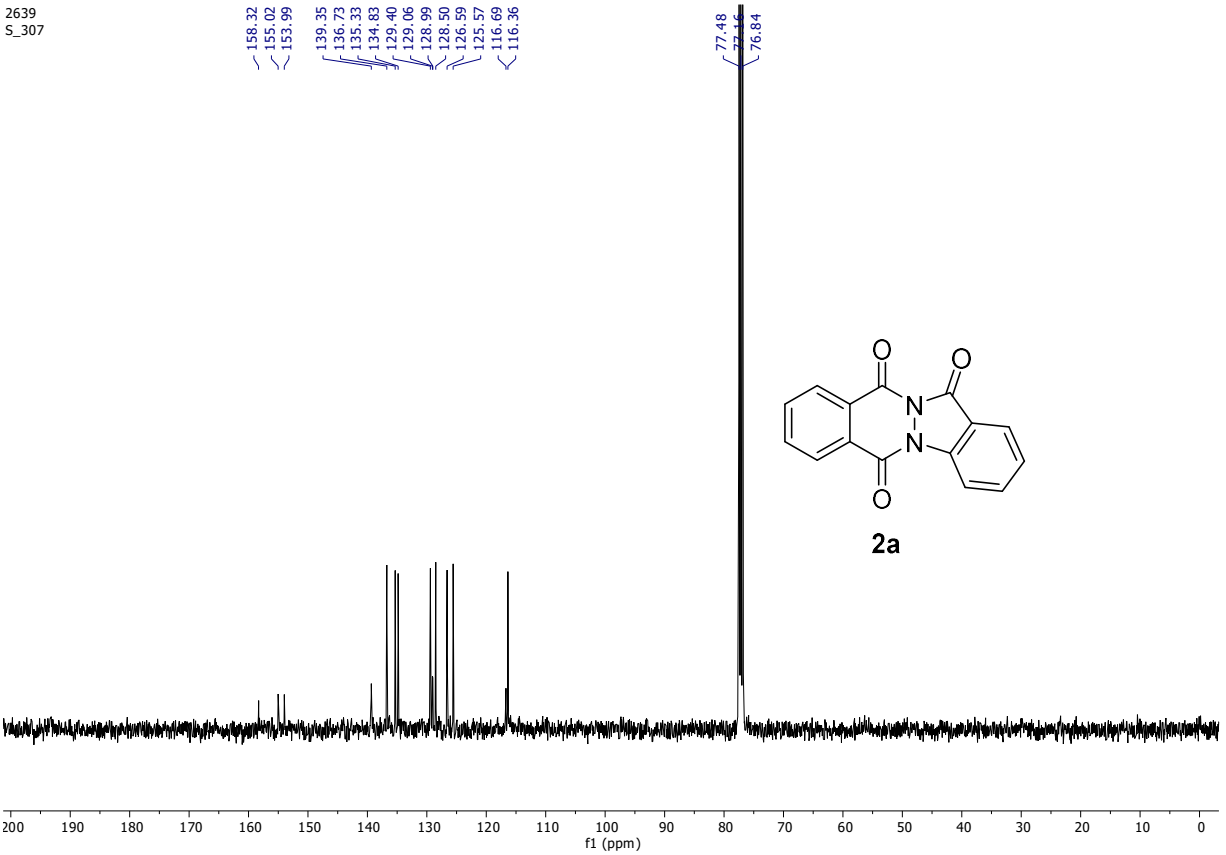
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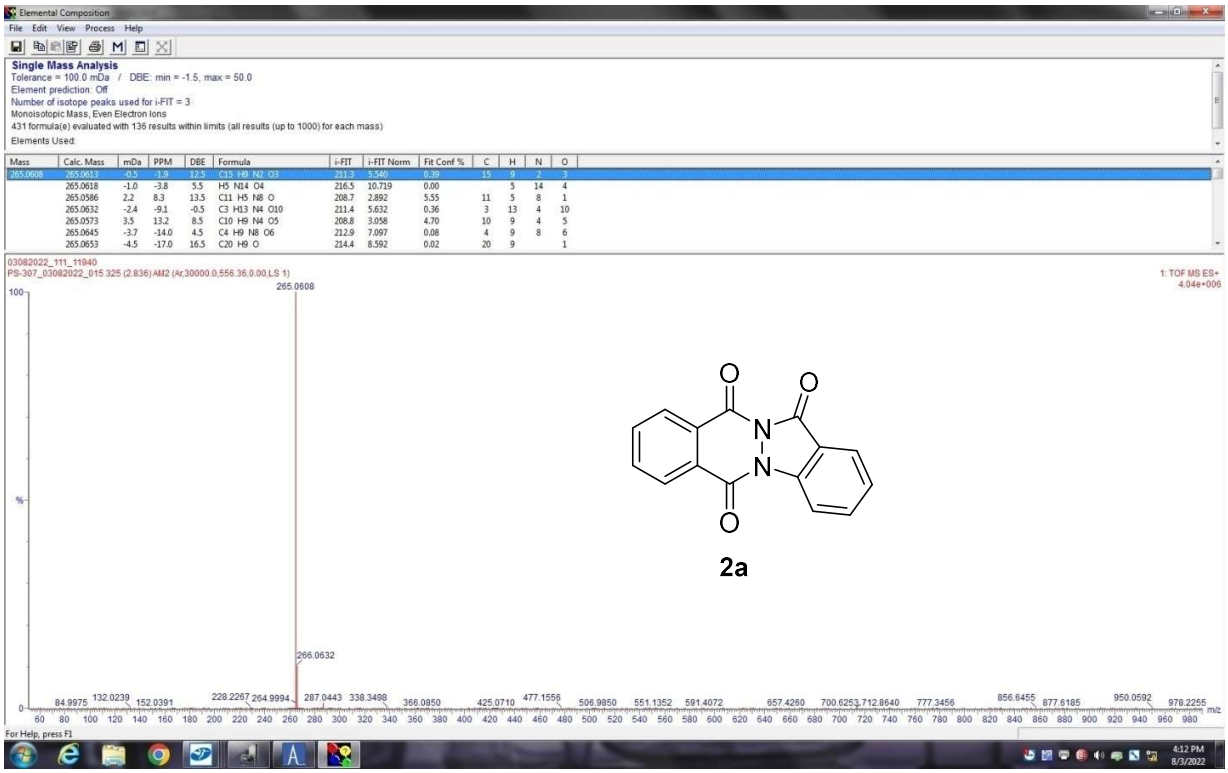
2a
f1 (ppm)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Number of isotope peaks used for i-FIT = 3
Monoisotopic Mass, Even Electron Ions
431 formula(e) evaluated with 136 results within limits (all results (up to 1000) for each mass)
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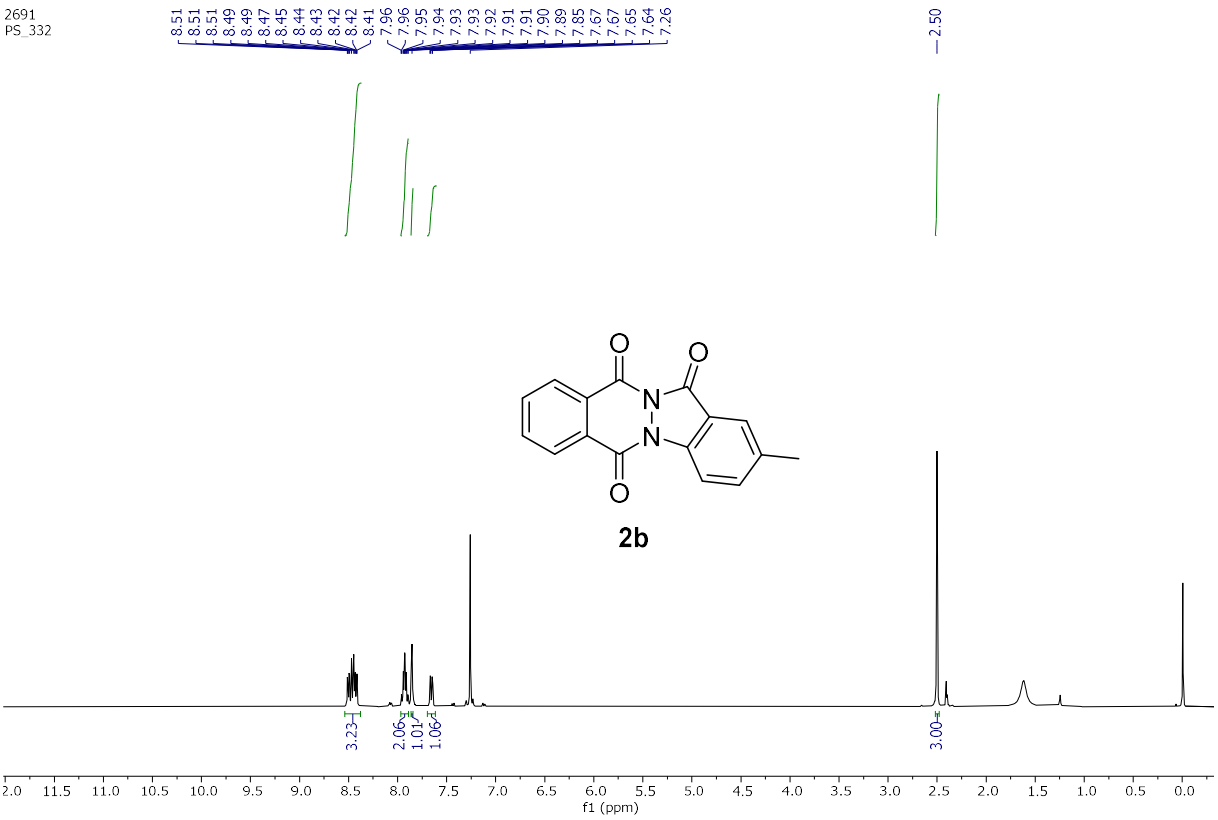
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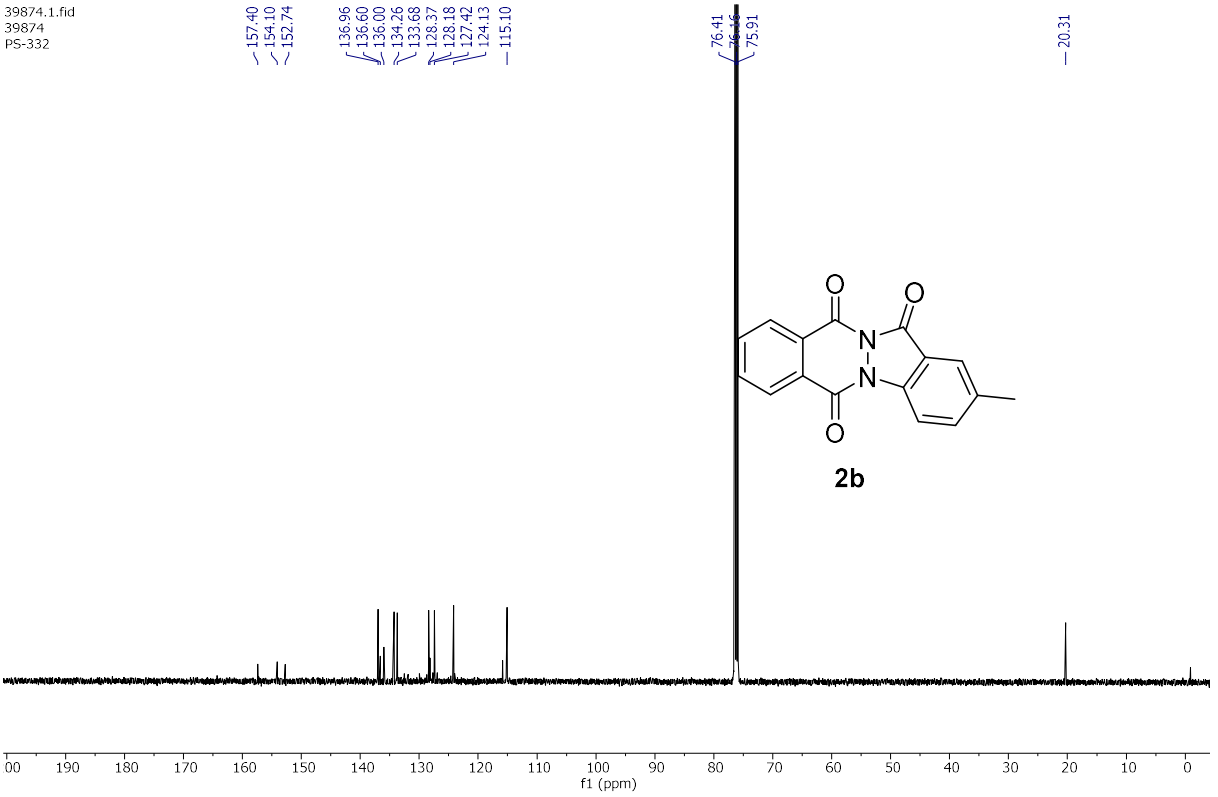
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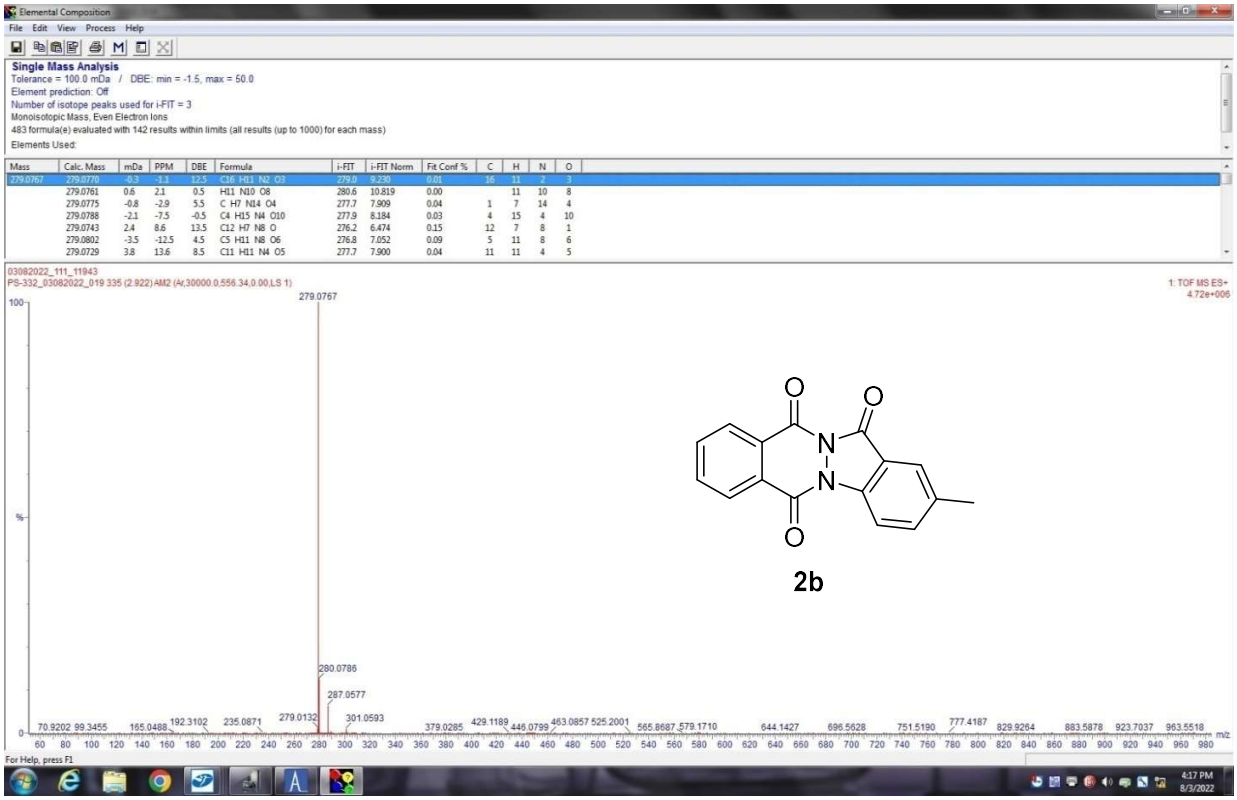
O
2b
f1 (ppm)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Monoisotopic Mass, Even Electron Ions
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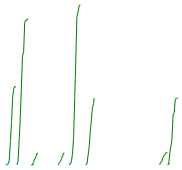

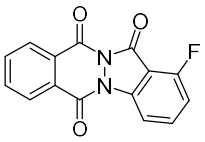

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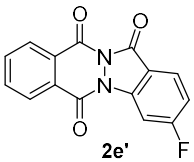

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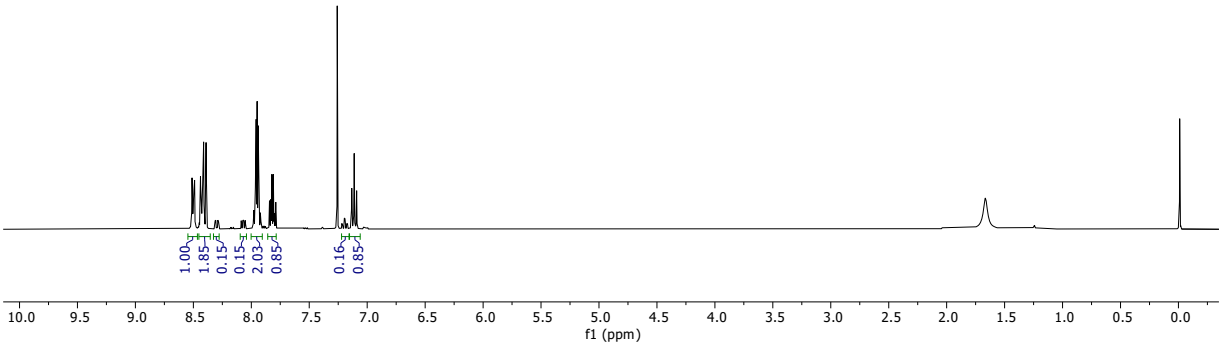
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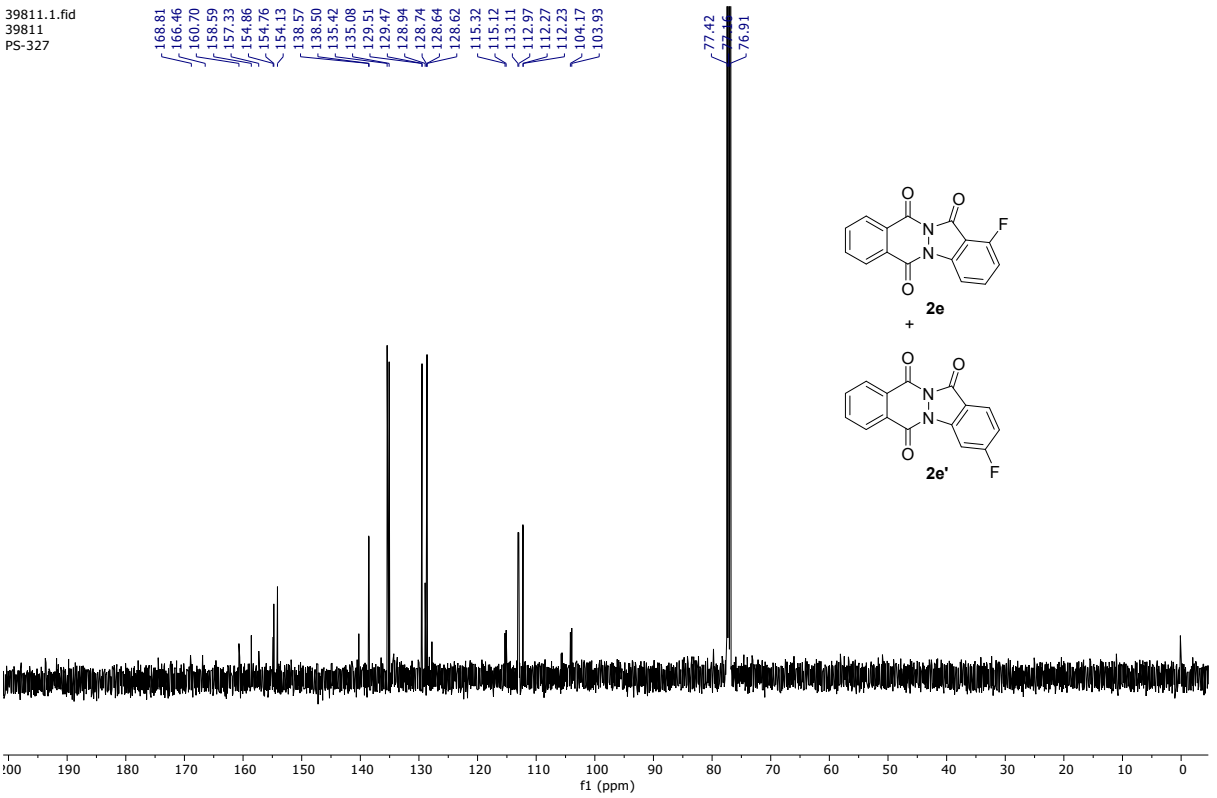
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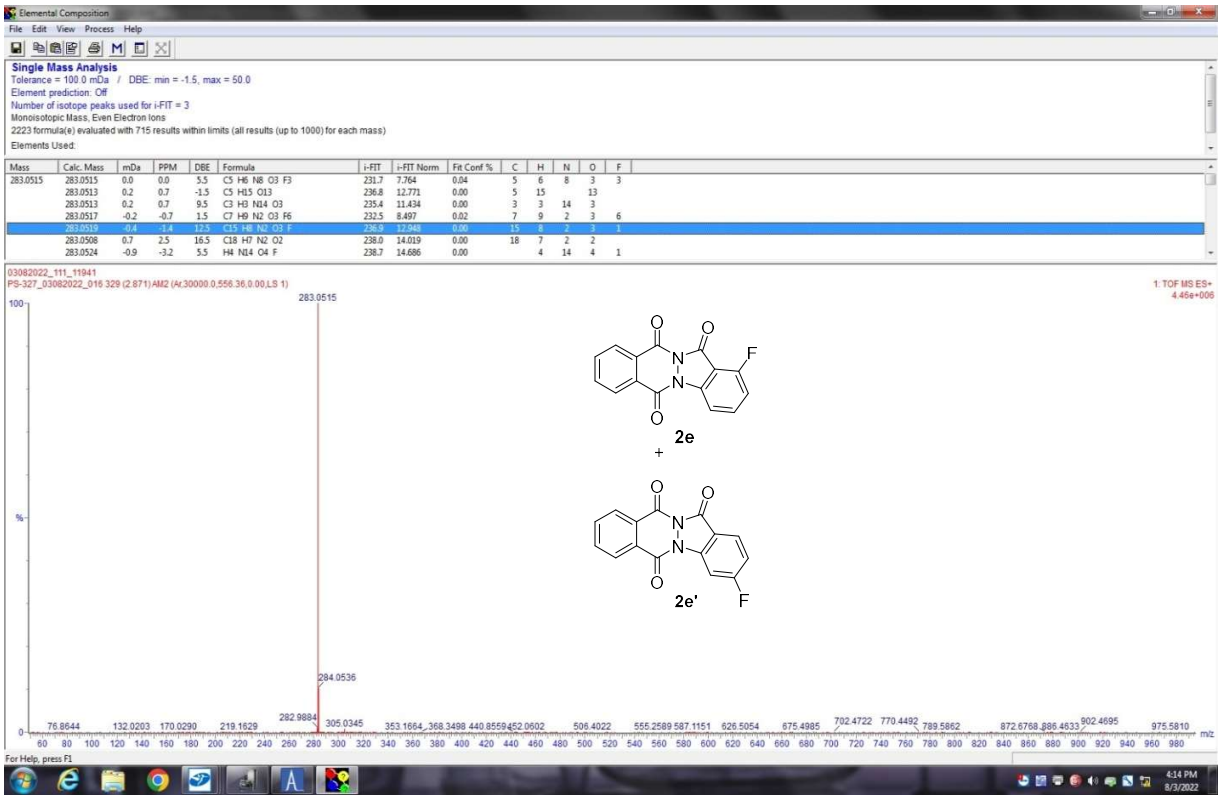
2e'
F
f1 (ppm)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Monoisotopic Mass, Even Electron Ions
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+
2e'
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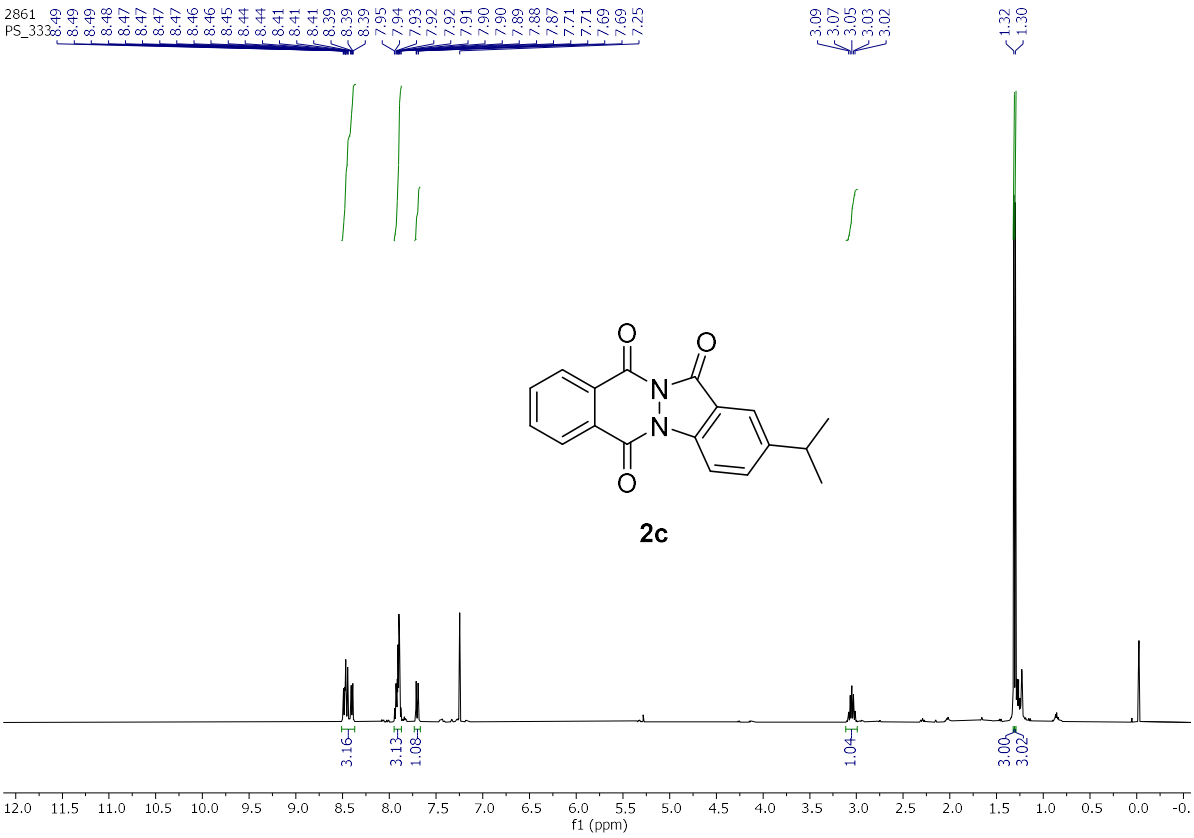
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3.09 3.07 3.05 3.03 3.02
1.32 1.30
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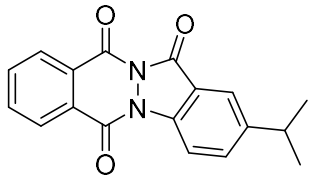
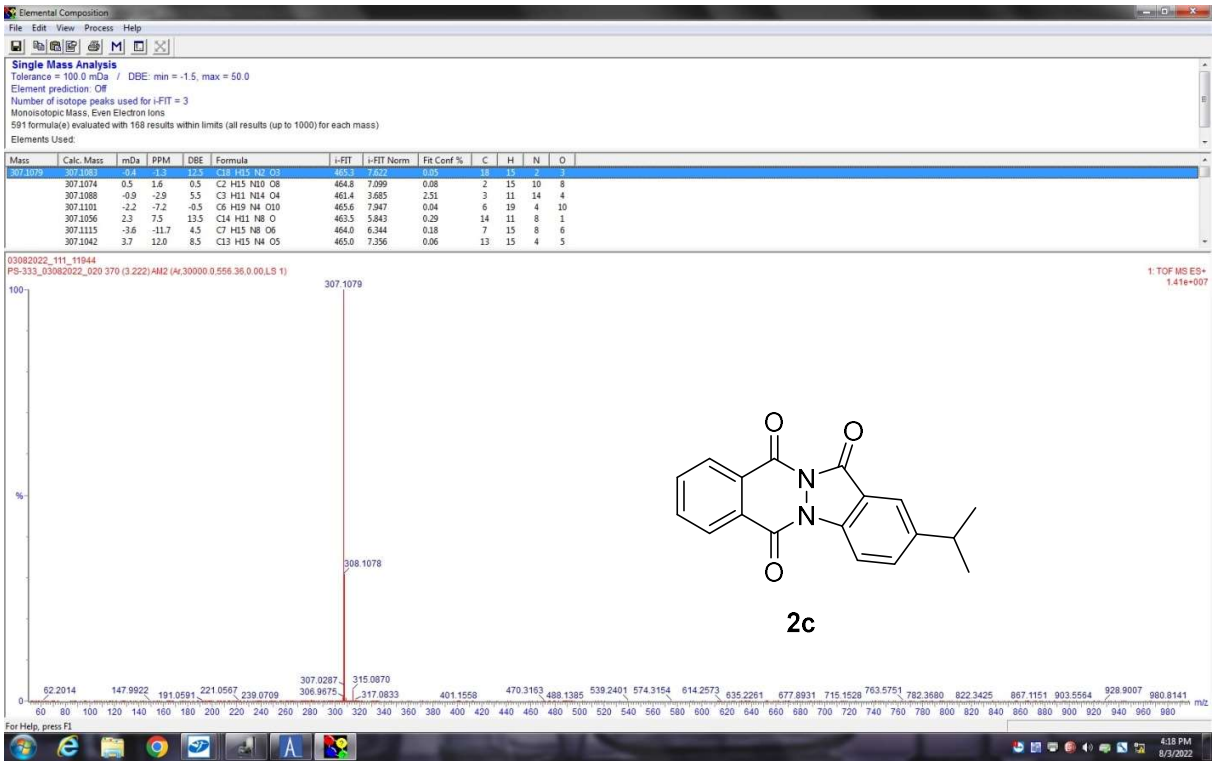
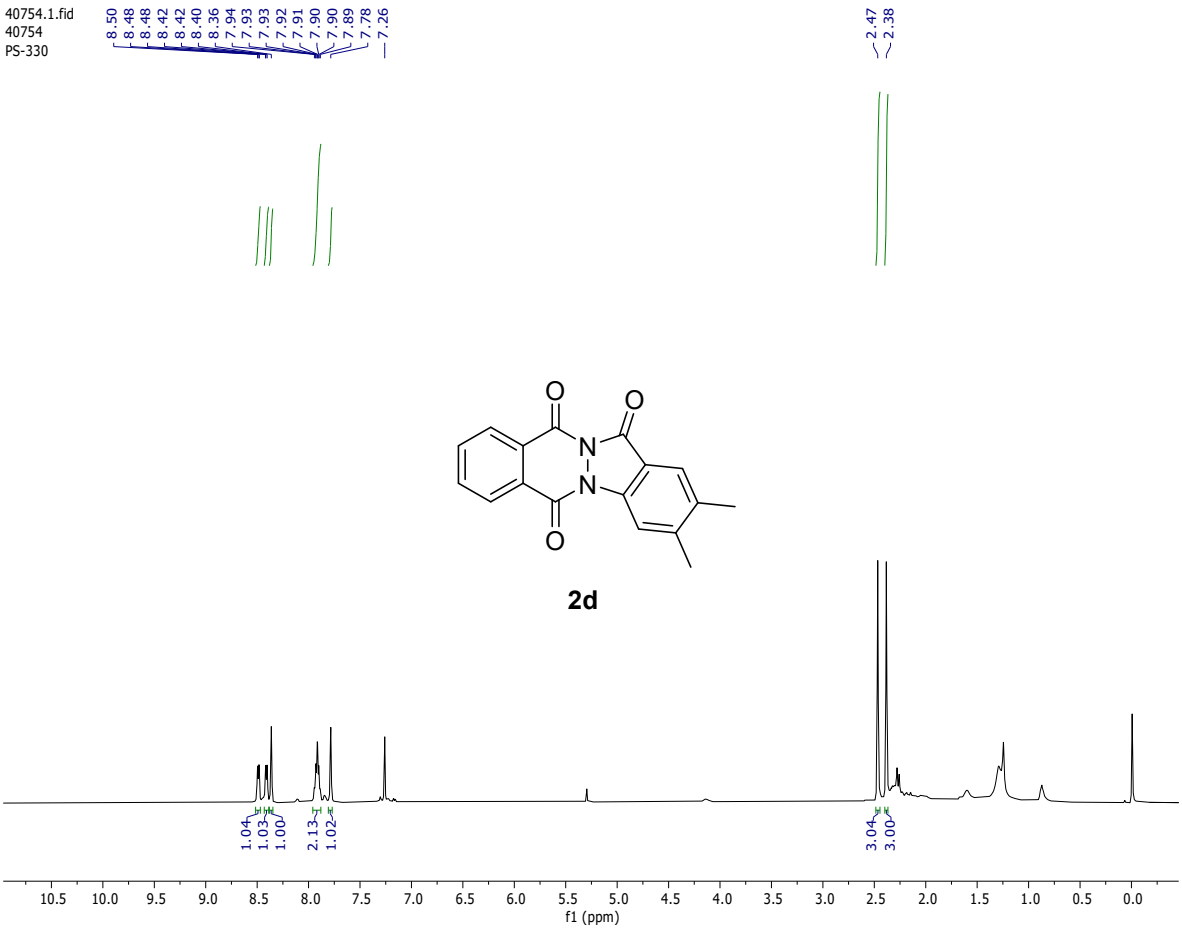
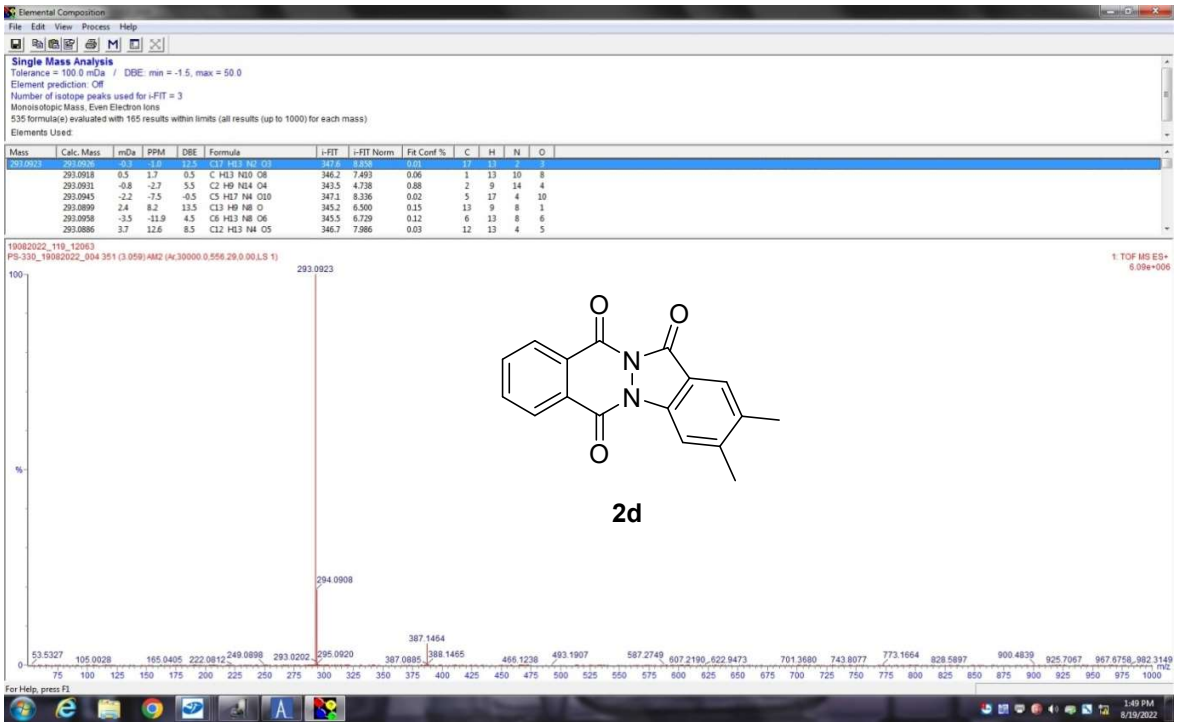


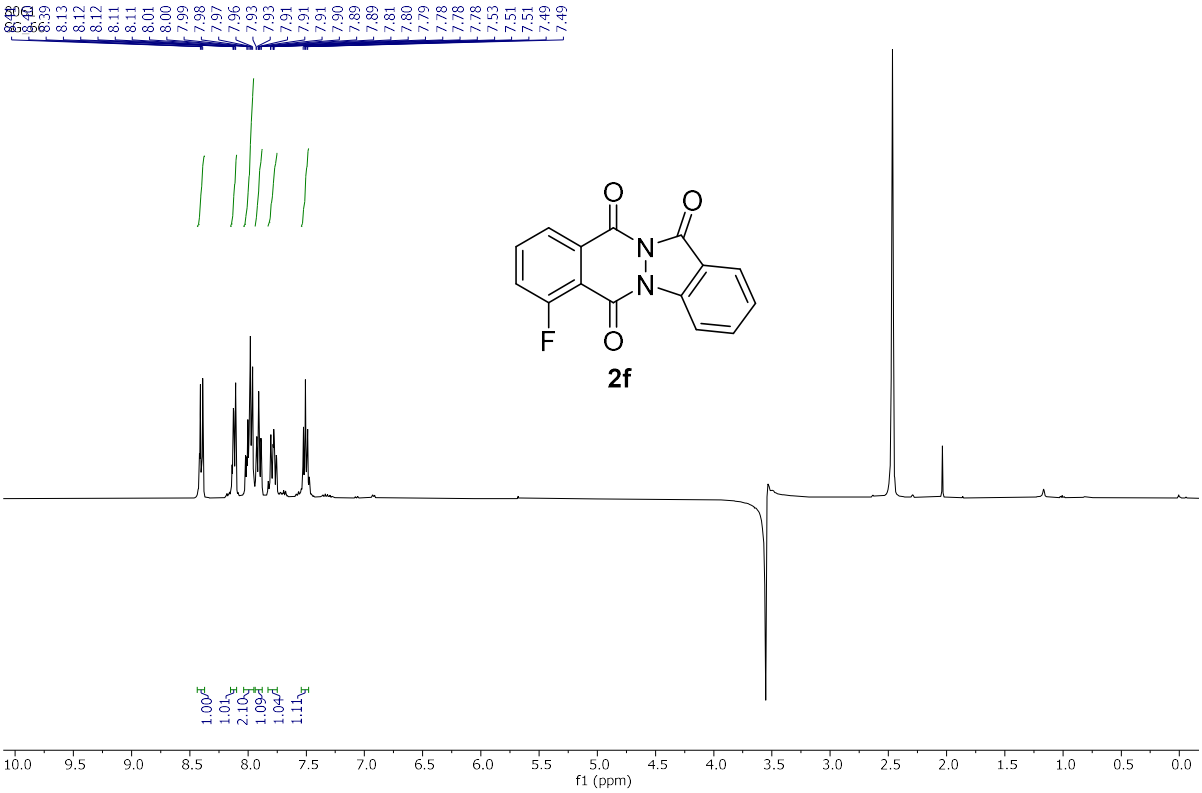
2c

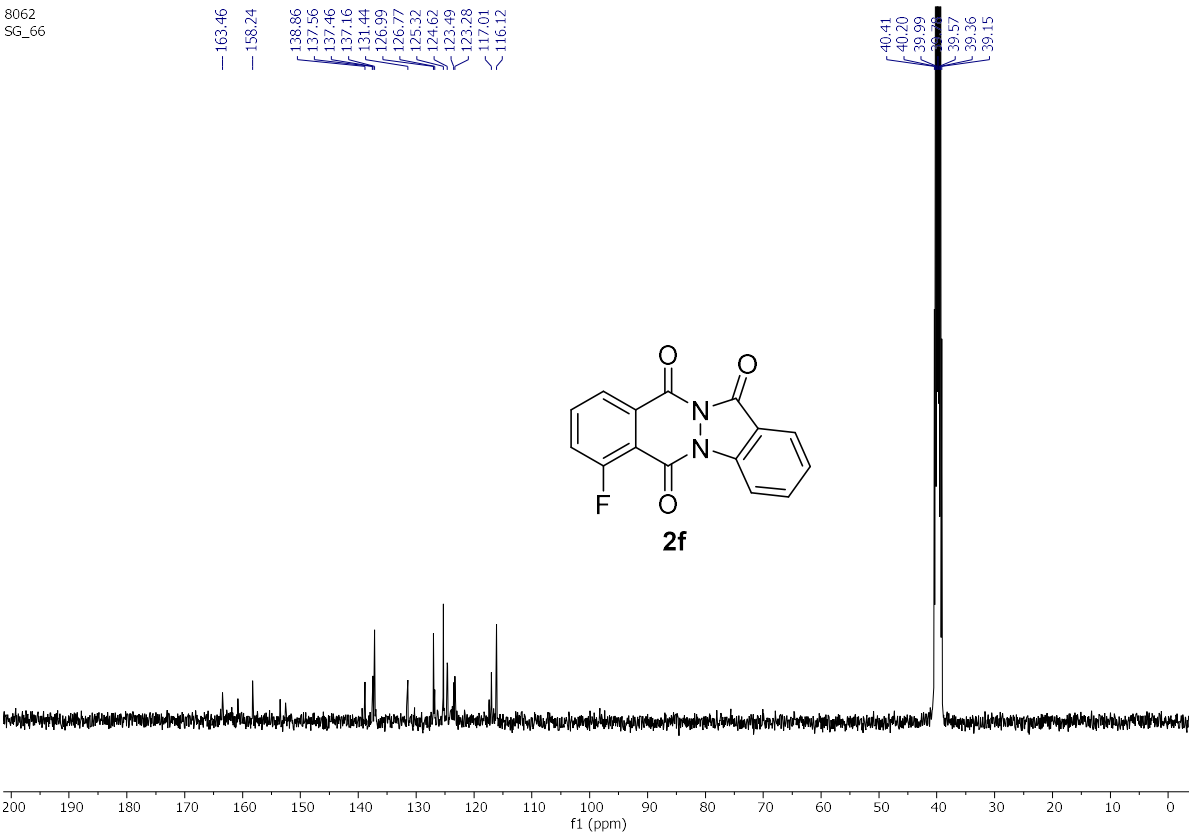


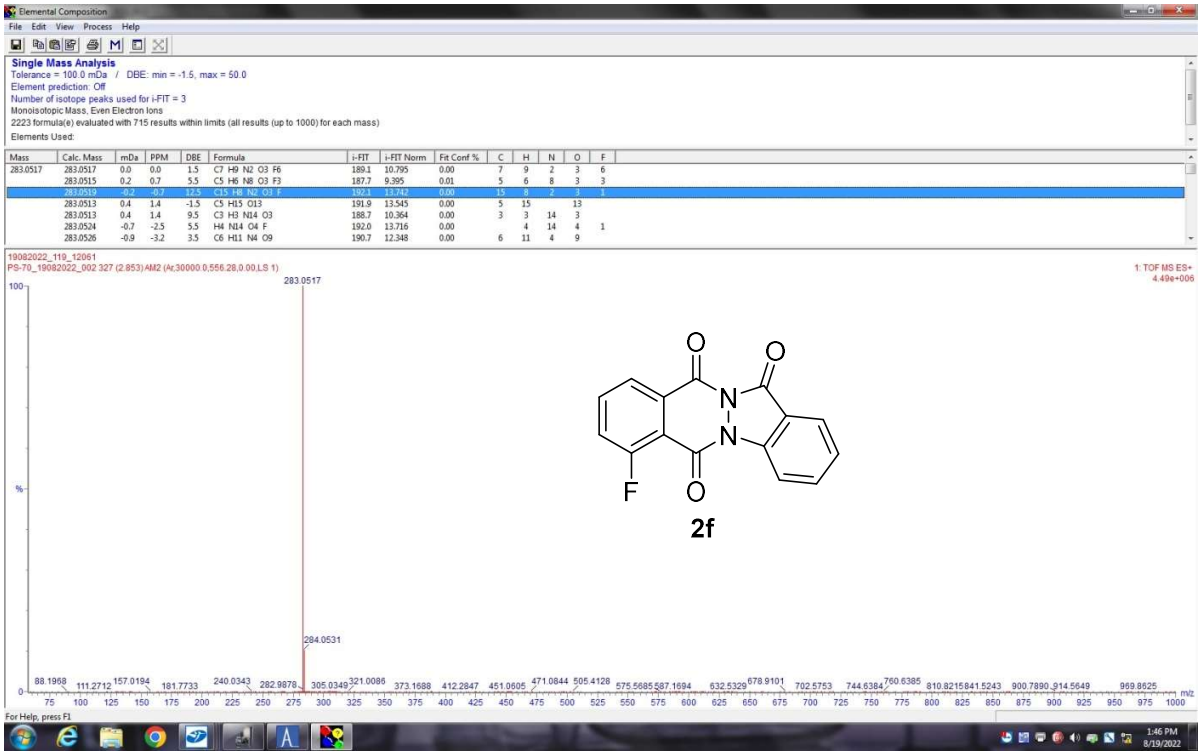
2c


40754.1.fid
40754
PS-330
8.50
8.48
8.48
8.42
8.42
8.40
8.36
7.94
7.93
7.93
7.92
7.91
7.90
7.90
7.89
7.78
7.26
2.47
2.38
O
O
N
N
O
2d
1.04
1.03
1.00
2.13
1.02
3.04
3.00
10.5
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)


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
535 formula(e) evaluated with 165 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
293.0923 293.0926 -0.3 -1.0 12.5 C17 H13 N2 O3 347.6 8.858 0.01 17 13 2 3
293.0918 0.5 1.7 0.5 C H13 N10 O8 346.2 7.493 0.06 1 13 10 8
293.0931 -0.8 -2.7 5.5 C2 H9 N14 O4 343.5 4.738 0.88 2 9 14 4
293.0945 -2.2 -7.5 -0.5 C5 H17 N4 O10 347.1 8.336 0.02 5 17 4 10
293.0899 2.4 8.2 13.5 C13 H9 N8 O 345.2 6.500 0.15 13 9 8 1
293.0958 -3.5 -11.9 4.5 C6 H13 N8 O6 345.5 6.729 0.12 6 13 8 6
293.0886 3.7 12.6 8.5 C12 H13 N4 O5 346.7 7.986 0.03 12 13 4 5
19082022_119_12063
PS-330_19082022_004 351 (3.059) AM2 (Ar,30000.0,556.29,0.00,LS 1)
1: TOF MS ES+
6.09e+006
293.0923
100
%
294.0908
387.1464
53.5327
105.0028
165.0405
222.0812
249.0898
293.0202
295.0920
387.0895
388.1465
466.1238
493.1907
587.2749
607.2190
622.9473
701.3680
743.8077
773.1664
828.5897
900.4839
925.7067
967.6758
982.3149
m/z
For Help, press F1
1:49 PM
8/19/2022
O
O
N
N
O
2d

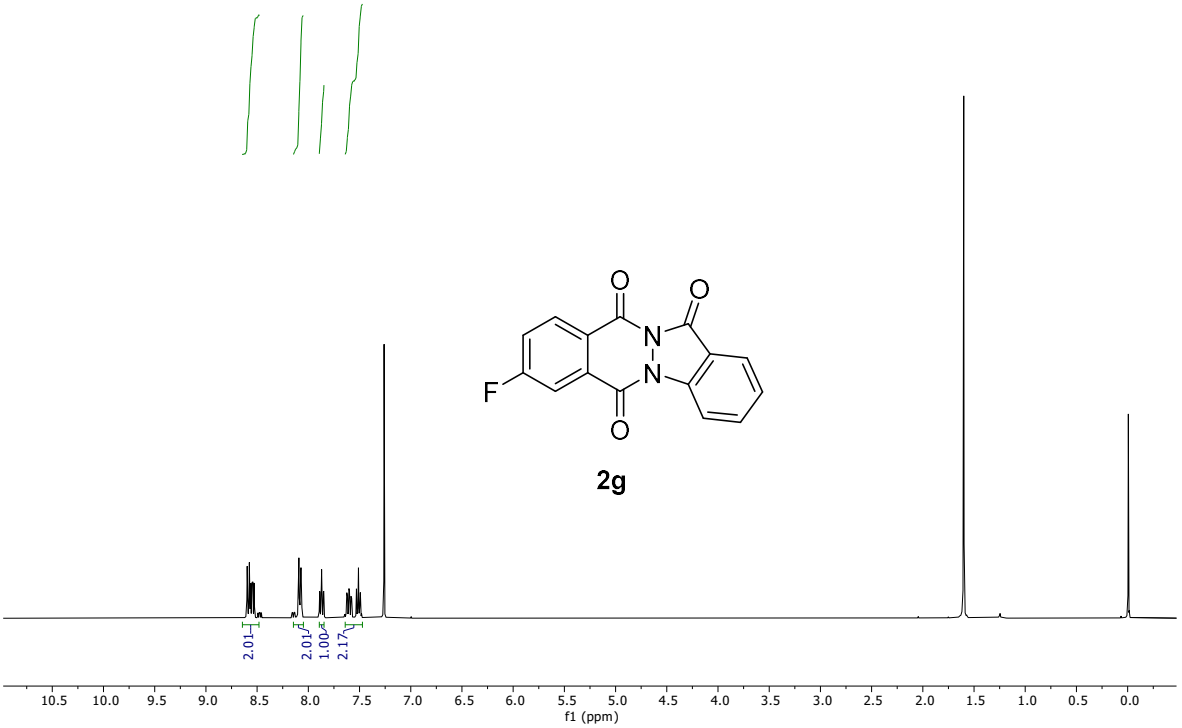

8.39
8.13
8.12
8.12
8.11
8.11
8.01
8.00
7.99
7.98
7.97
7.96
7.93
7.93
7.91
7.91
7.90
7.89
7.89
7.81
7.80
7.79
7.78
7.78
7.78
7.53
7.51
7.51
7.49
7.49
1.00
1.01
2.10
1.09
1.04
1.11
O
O
N
N
O
F
2f
f1 (ppm)


8062
SG_66
163.46
158.24
138.86
137.56
137.46
137.16
131.44
126.99
126.77
125.32
124.62
123.49
123.28
117.01
116.12
40.41
40.20
39.99
39.57
39.36
39.15
O
O
N
N
O
F
2f
f1 (ppm)

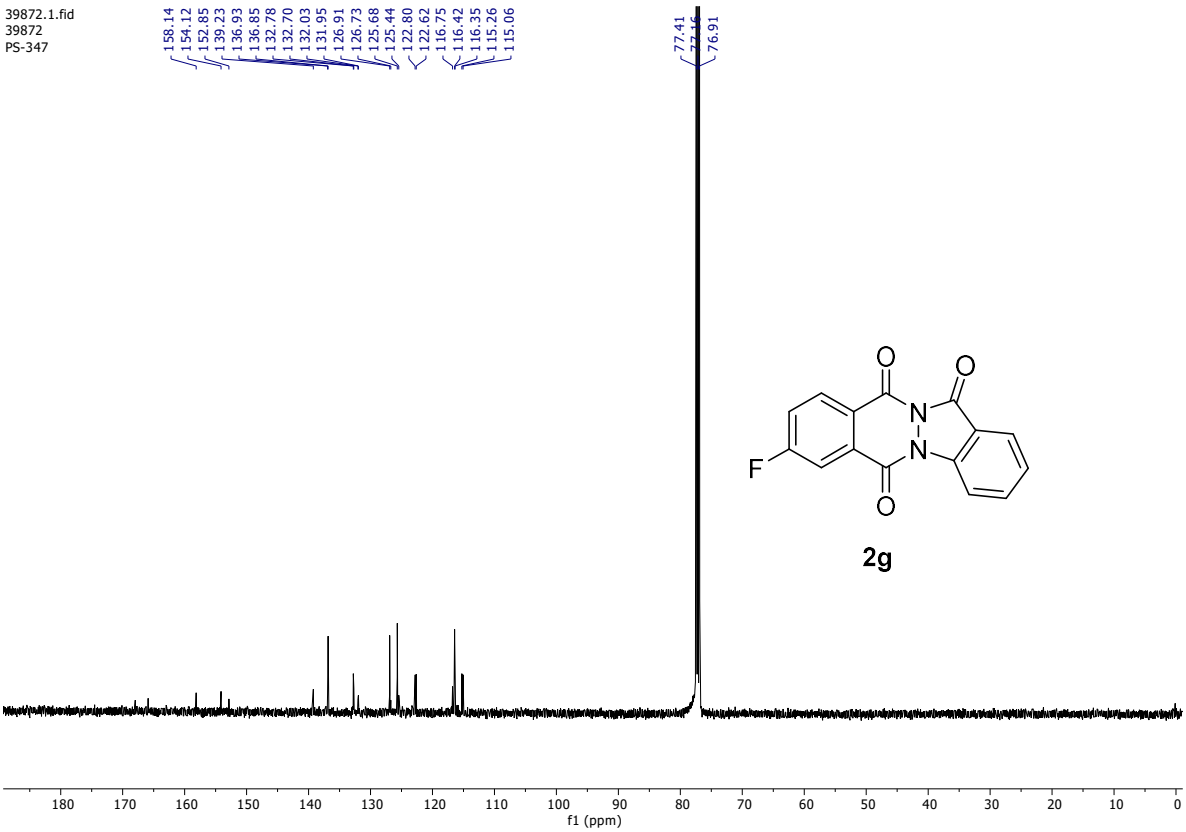

Elemental Composition
Single Mass Analysis
Tolerance = 100.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3
Monoisotopic Mass, Even Electron Ions
2223 formula(e) evaluated with 715 results within limits (all results (up to 1000) for each mass)
Elements Used:
283.0517
284.0531
1: TOF MS ES+
4.49e+006

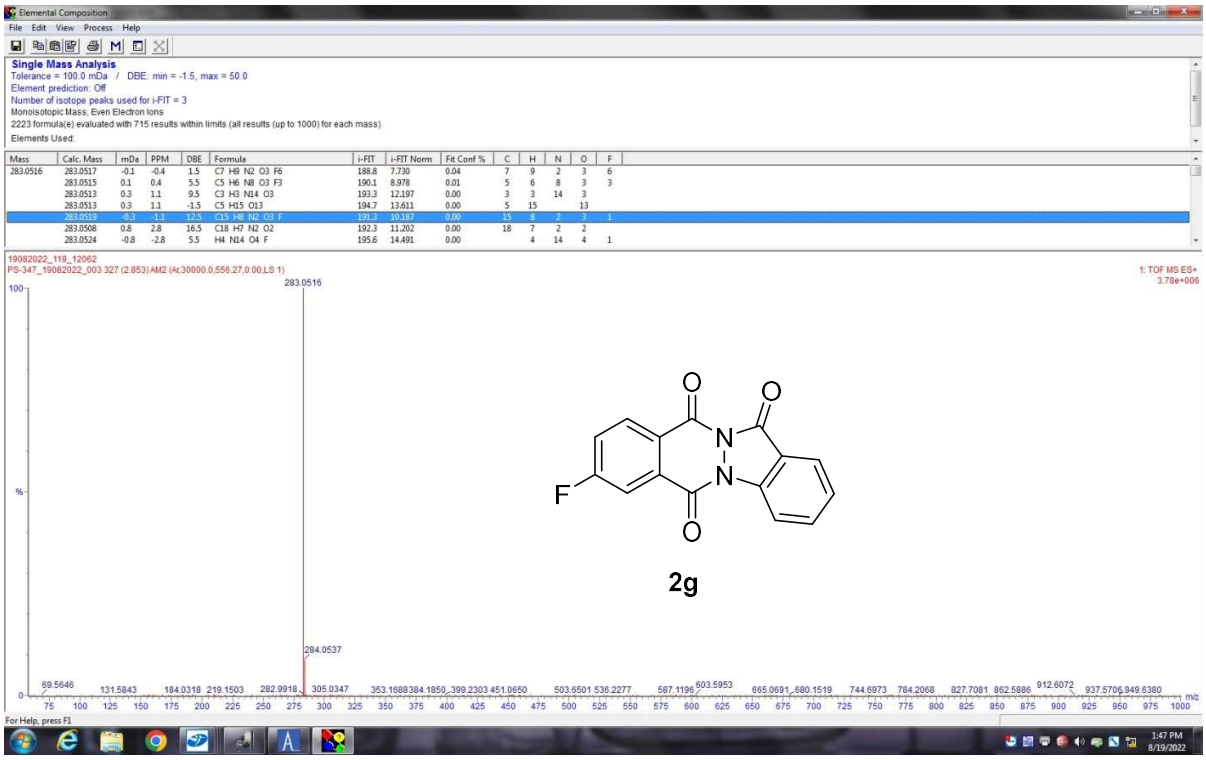
2f

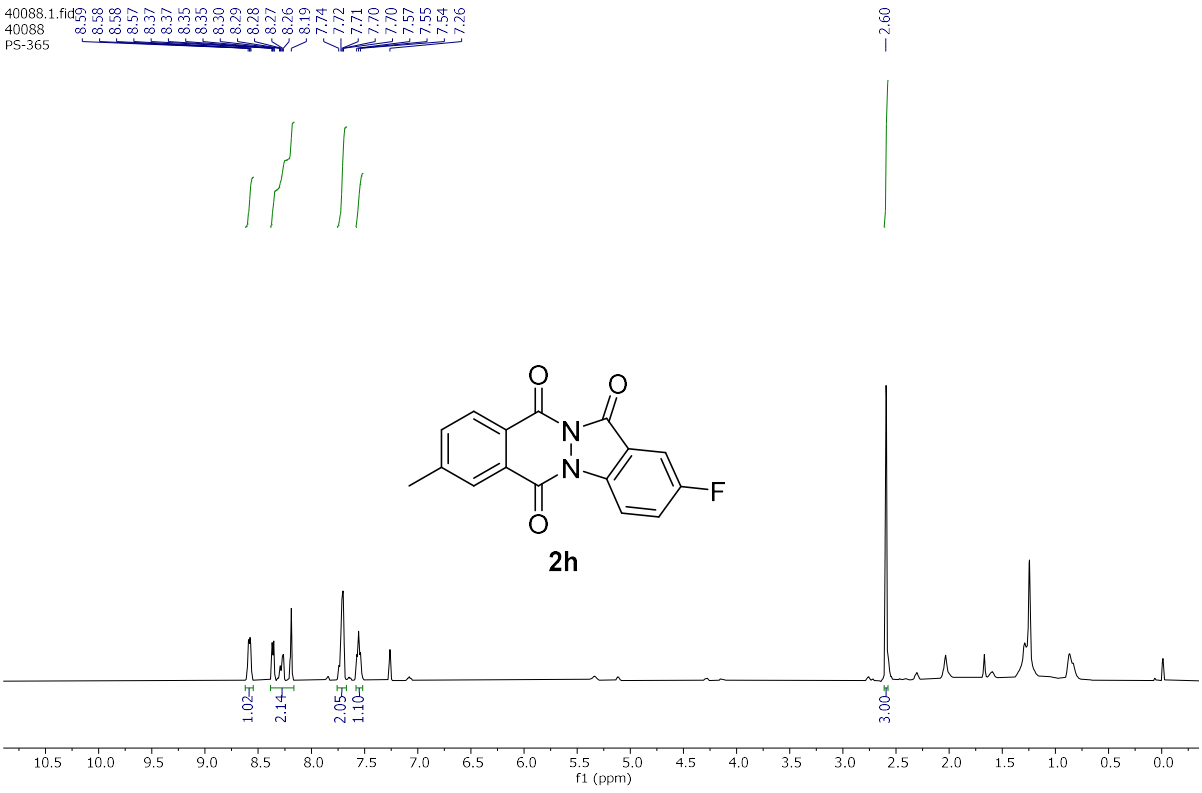

2677
PS_347

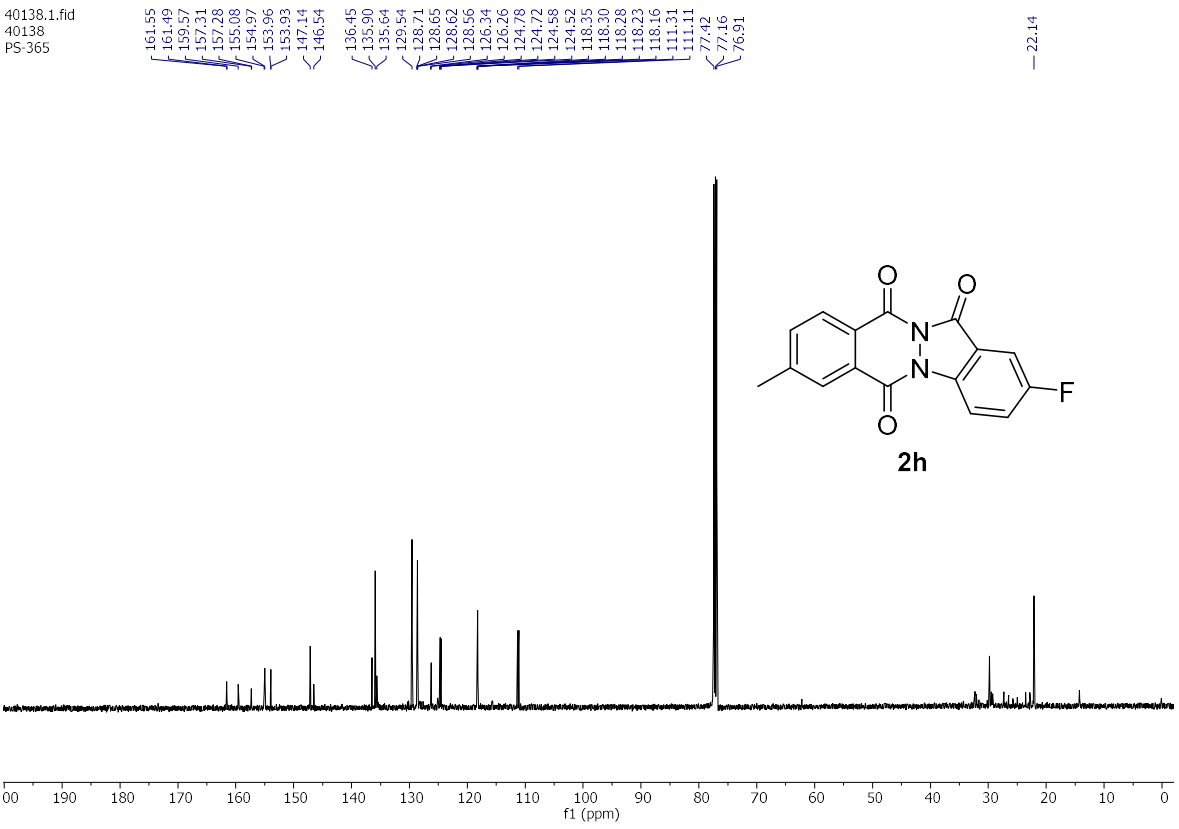

2.01
2.01
1.00
2.17
1
f1 (ppm)

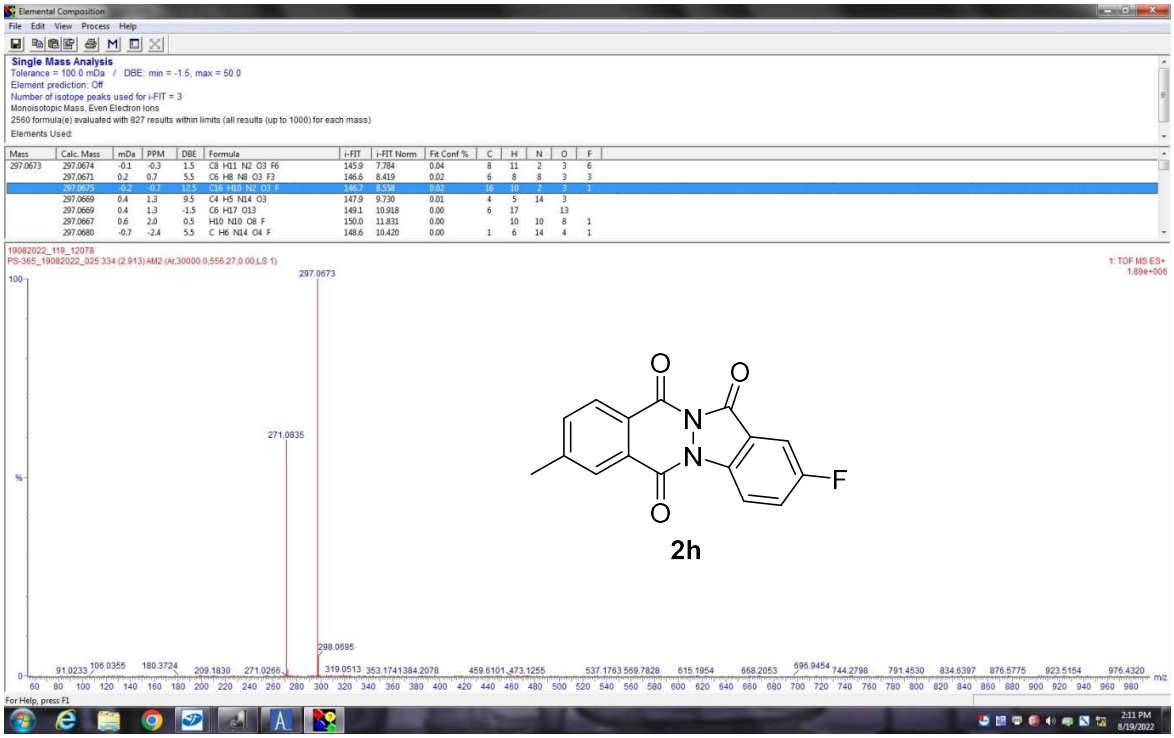
2g

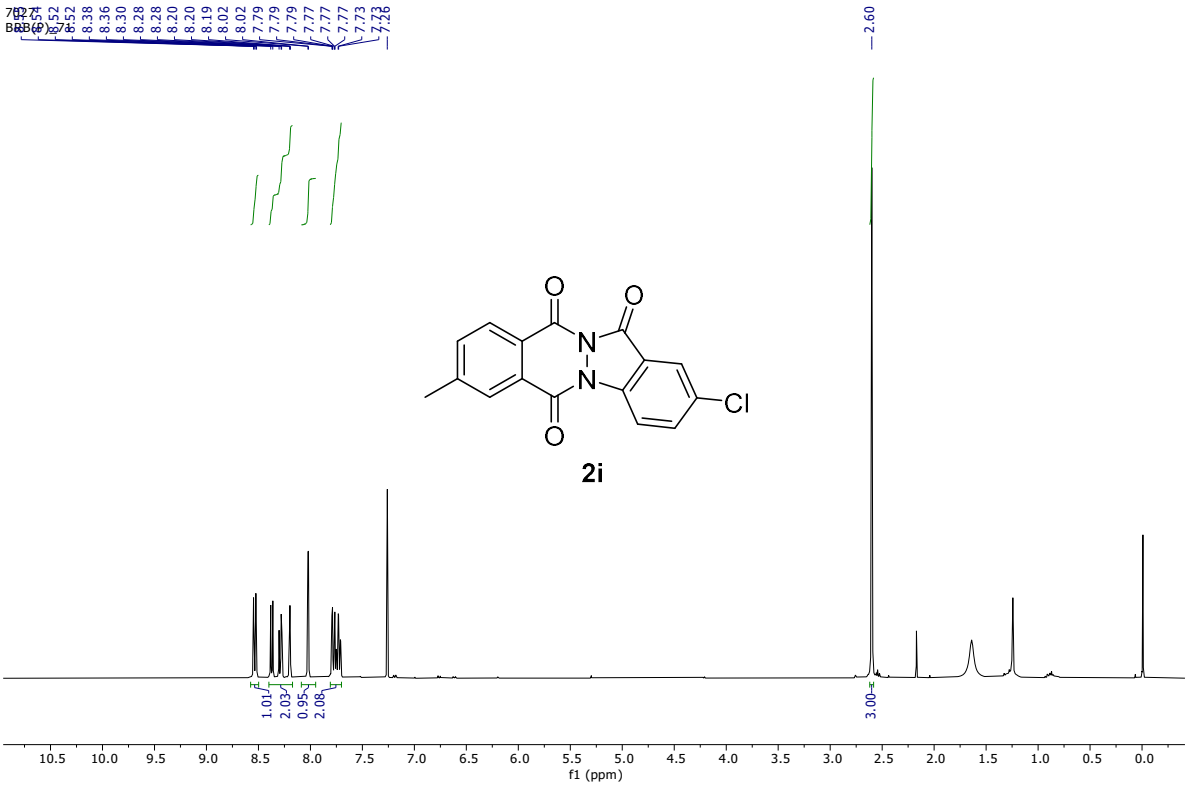

39872.1.fid
39872
PS-347
158.14
154.12
152.85
139.23
136.93
136.85
132.78
132.70
132.03
131.95
126.91
126.73
125.68
125.44
122.80
122.62
116.75
116.42
116.35
115.26
115.06
77.41
77.16
76.91
F
2g
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

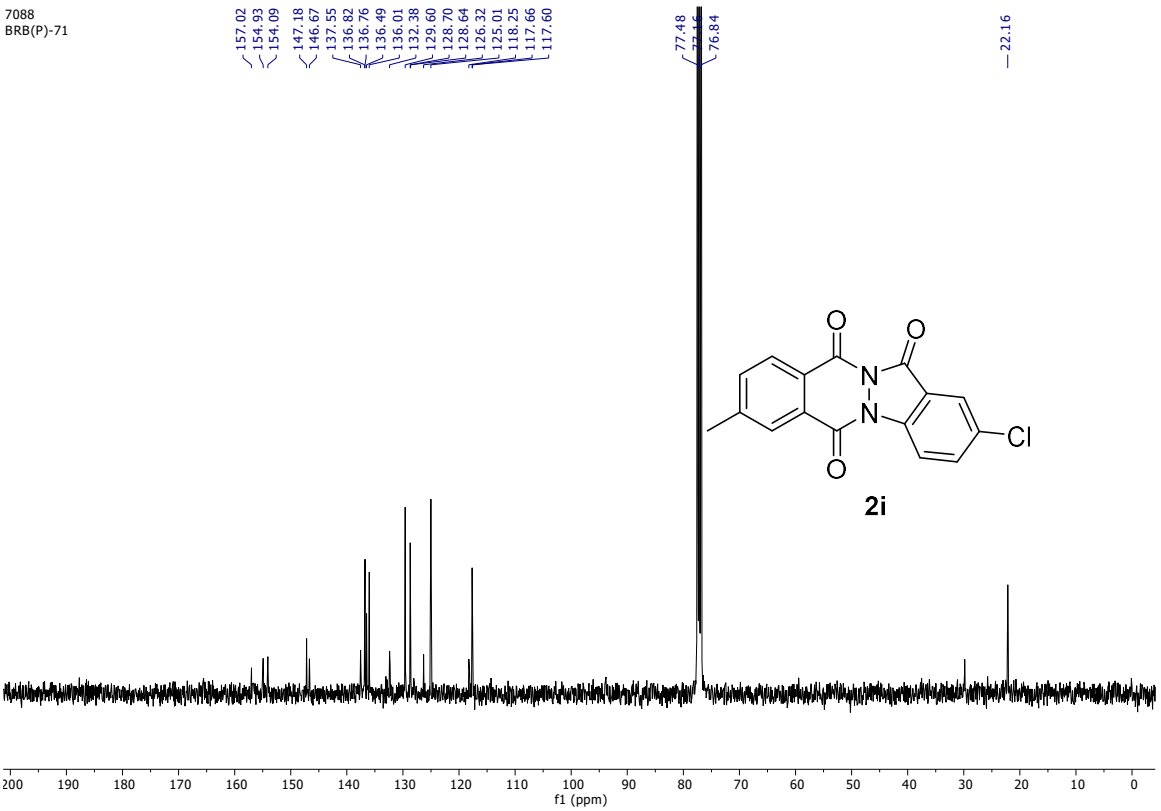

Elemental Composition
File Edit View Process Help
Single Mass Analysis
Tolerance = 100.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3
Monoisotopic Mass, Even Electron Ions
2223 formula(e) evaluated with 715 results within limits (all results (up 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
283.0516 283.0517 -0.1 -0.4 1.5 C7 H9 N2 O3 F6 188.8 7.730 0.04 7 9 2 3 6
283.0515 0.1 0.4 5.5 C5 H6 N8 O3 F3 190.1 8.978 0.01 5 6 8 3 3
283.0513 0.3 1.1 9.5 C3 H3 N14 O3 194.3 12.197 0.00 3 3 14 3
283.0513 0.3 1.1 -1.5 C5 H15 O13 194.7 13.611 0.00 5 15 13
283.0519 -0.3 -1.1 12.5 C15 H8 N2 O3 F 191.3 10.187 0.00 15 8 2 3 1
283.0508 0.8 2.8 16.5 C18 H7 N2 O2 192.3 11.202 0.00 18 7 2 2
283.0524 -0.8 -2.8 5.5 H4 N14 O4 F 195.6 14.491 0.00 4 14 4 1
19082022_119_12062
PS-347_19082022_003 327 (2.853) AM2 (Ar,30000.0,556.27,0.00,LS 1)
1: TOF MS ES+
3.78e+006
283.0516
284.0537
F
2g
For Help, press F1
1:47 PM
8/19/2022

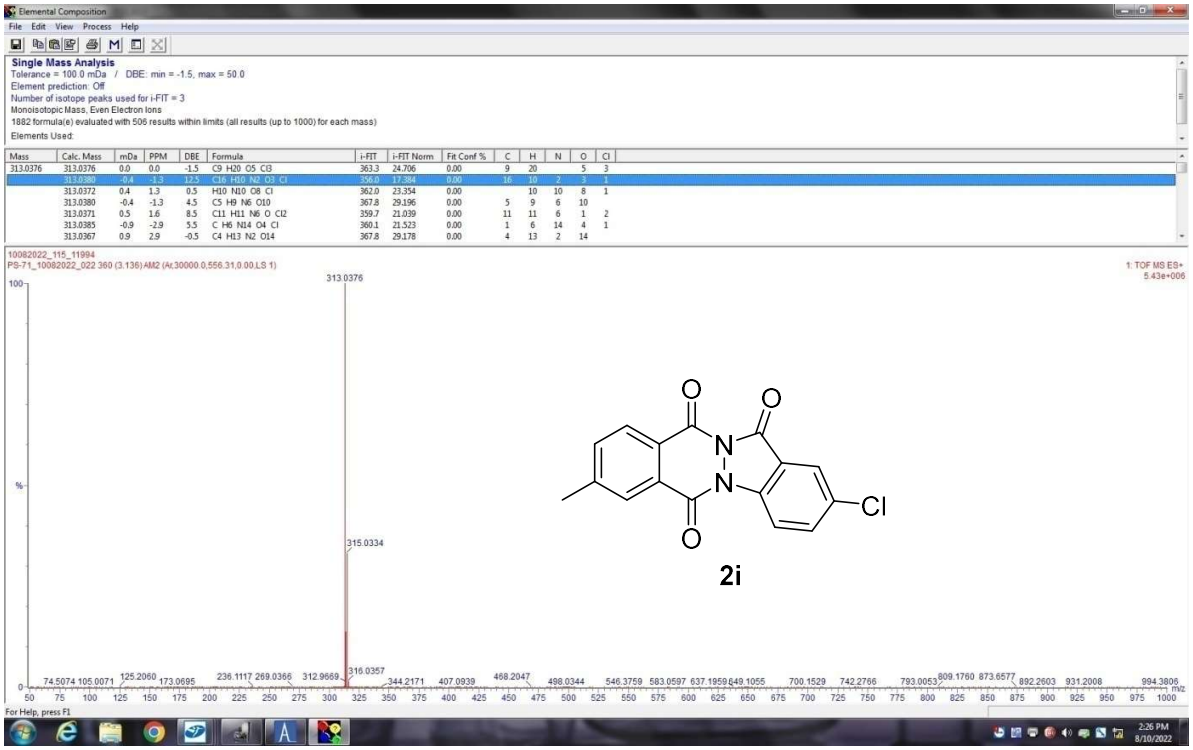

40088.1.fid
40088
PS-365
8.39
8.38
8.38
8.37
8.37
8.37
8.35
8.35
8.30
8.29
8.28
8.27
8.26
8.19
7.74
7.72
7.71
7.70
7.70
7.57
7.55
7.54
7.26
2.60
O
O
N
N
O
F
2h
1.02
2.14
2.05
1.10
3.00
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

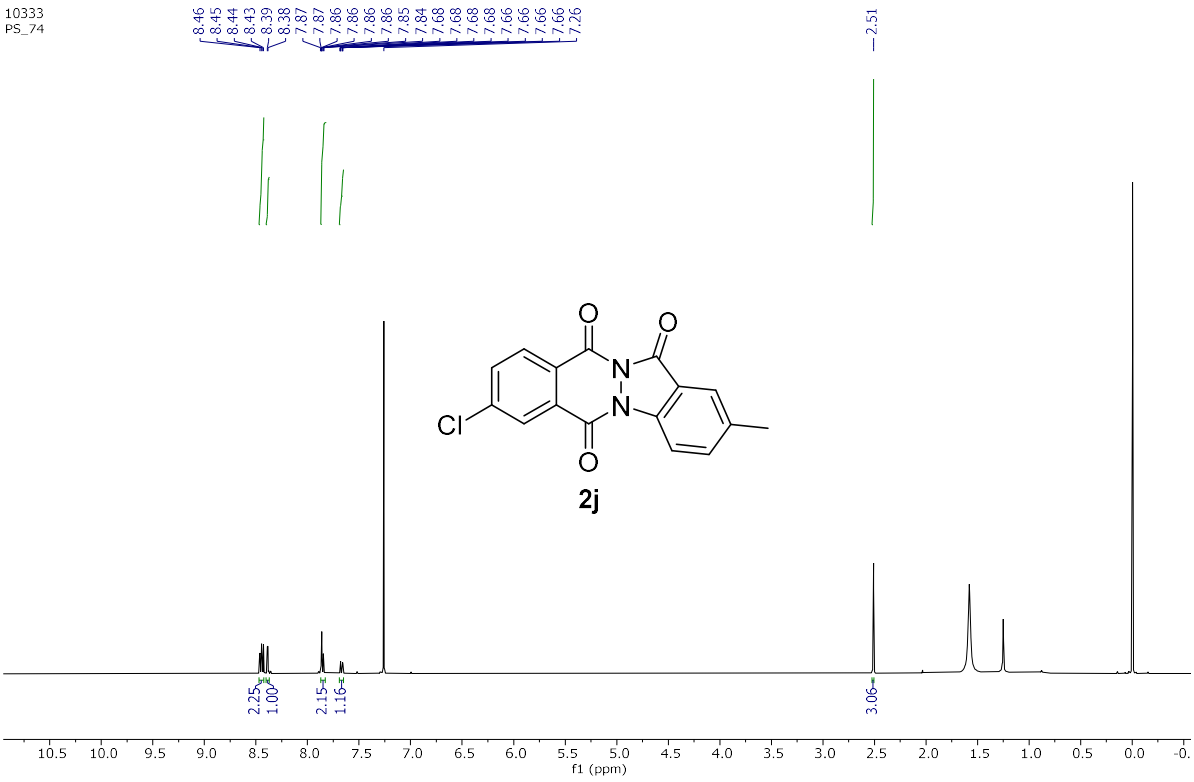

40138.1.fid
40138
PS-365
161.55
161.49
159.57
157.31
157.28
155.08
154.97
153.96
153.93
147.14
146.54
136.45
135.90
135.64
129.54
128.71
128.65
128.62
128.56
126.34
126.26
124.78
124.72
124.58
124.52
118.35
118.30
118.28
118.23
118.16
111.31
111.11
77.42
77.16
76.91
22.14
O
O
N
N
O
F
2h
00 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

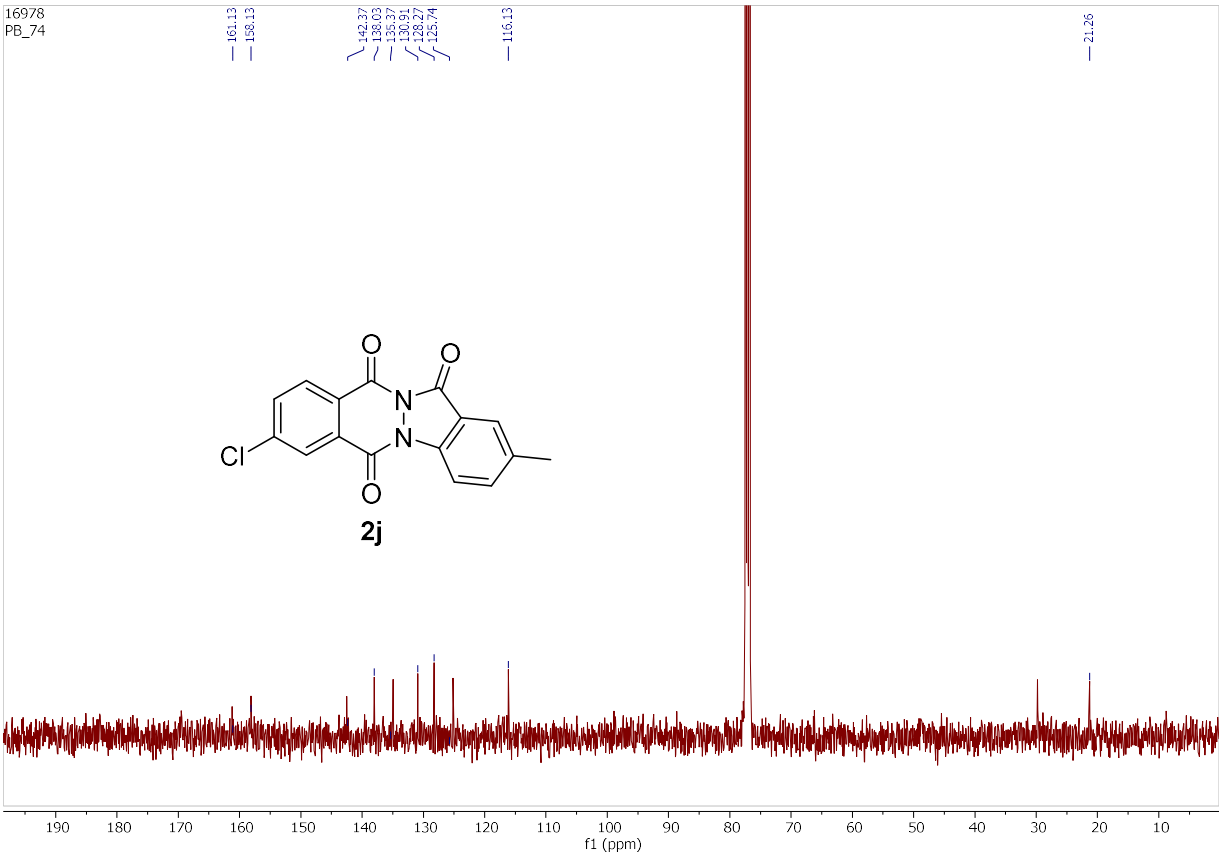

Elemental Composition
Single Mass Analysis
Tolerance = 100.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3
Monoisotopic Mass, Even Electron Ions
2560 formula(e) evaluated with 827 results within limits (all results (up to 1000) for each mass)
Elements Used:
297.0673
271.0835
298.0695
2h

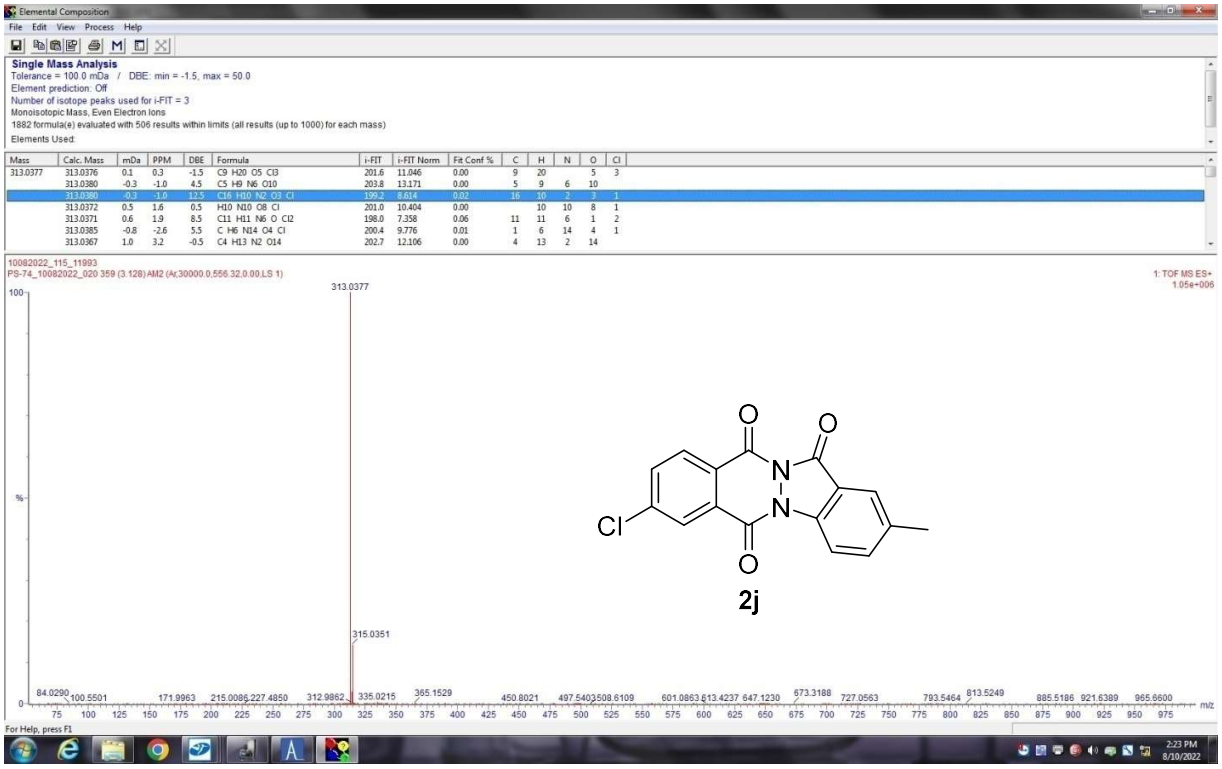

2.60
2i
1.01
2.03
0.95
2.08
3.00
f1 (ppm)


7088
BRB(P)-71
157.02
154.93
154.09
147.18
146.67
137.55
136.82
136.76
136.49
136.01
132.38
129.60
128.70
128.64
126.32
125.01
118.25
117.66
117.60
77.48
77.16
76.84
22.16
O
O
N
N
O
Cl
2i
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

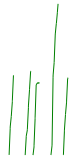

Elemental Composition
File Edit View Process Help
Single Mass Analysis
Tolerance = 100.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3
Monoisotopic Mass, Even Electron Ions
1882 formula(e) evaluated with 506 results within limits (all results (up 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
313.0376 | 313.0376 | 0.0 | 0.0 | -1.5 | C9 H20 O5 Cl3 | 363.3 | 24.706 | 0.00 | 9 | 20 | | 5 | 3
313.0380 | -0.4 | -1.3 | 12.5 | C16 H10 N2 O3 Cl | 356.0 | 17.384 | 0.00 | 16 | 10 | 2 | 3 | 1
313.0372 | 0.4 | 1.3 | 0.5 | H10 N10 O8 Cl | 362.0 | 23.354 | 0.00 | | 10 | 10 | 8 | 1
313.0380 | -0.4 | -1.3 | 4.5 | C5 H9 N6 O10 | 367.8 | 29.196 | 0.00 | 5 | 9 | 6 | 10 |
313.0371 | 0.5 | 1.6 | 8.5 | C11 H11 N6 O Cl2 | 359.7 | 21.039 | 0.00 | 11 | 11 | 6 | 1 | 2
313.0385 | -0.9 | -2.9 | 5.5 | C H6 N14 O4 Cl | 360.1 | 21.523 | 0.00 | 1 | 6 | 14 | 4 | 1
313.0367 | 0.9 | 2.9 | -0.5 | C4 H13 N2 O14 | 367.8 | 29.178 | 0.00 | 4 | 13 | 2 | 14 |
10082022_115_11994
PS-71_10082022_022 360 (3.136) AM2 (Ar,30000.0,556.31,0.00,LS 1)
1: TOF MS ES+
5.43e+006
100
%
313.0376
315.0334
316.0357
0
74.5074 105.0071 125.2060 173.0695 236.1117 269.0366 312.9669 344.2171 407.0939 468.2047 498.0344 546.3759 583.0597 637.1959 649.1055 700.1529 742.2766 793.0053 809.1760 873.6577 892.2603 931.2008 994.3806
50 75 100 125 150 175 200 225 250 275 300 325 350 375 400 425 450 475 500 525 550 575 600 625 650 675 700 725 750 775 800 825 850 875 900 925 950 975 1000
m/z
For Help, press F1
O
O
N
N
O
Cl
2i
2:26 PM
8/10/2022

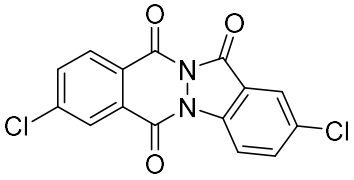

10333
PS_74
8.46
8.45
8.44
8.43
8.39
8.38
7.87
7.87
7.86
7.86
7.86
7.86
7.85
7.84
7.68
7.68
7.68
7.66
7.66
7.66
7.66
7.26
2.51
Cl
O
O
N
N
O
2j
2.25
1.00
2.15
1.16
3.06
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.
f1 (ppm)


16978
PB_74
161.13
158.13
142.37
138.03
135.37
130.91
128.27
125.74
116.13
21.26
Cl
O
O
N
N
O
2j
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

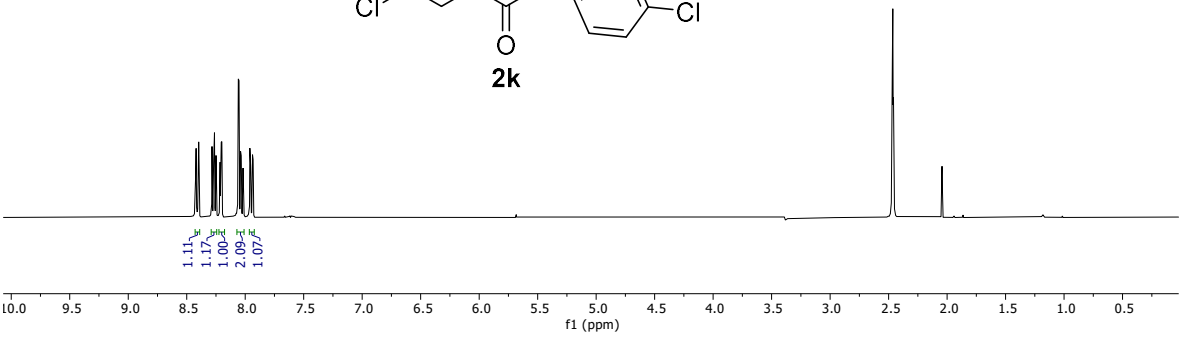

Elemental Composition
Single Mass Analysis
313.0377
315.0351

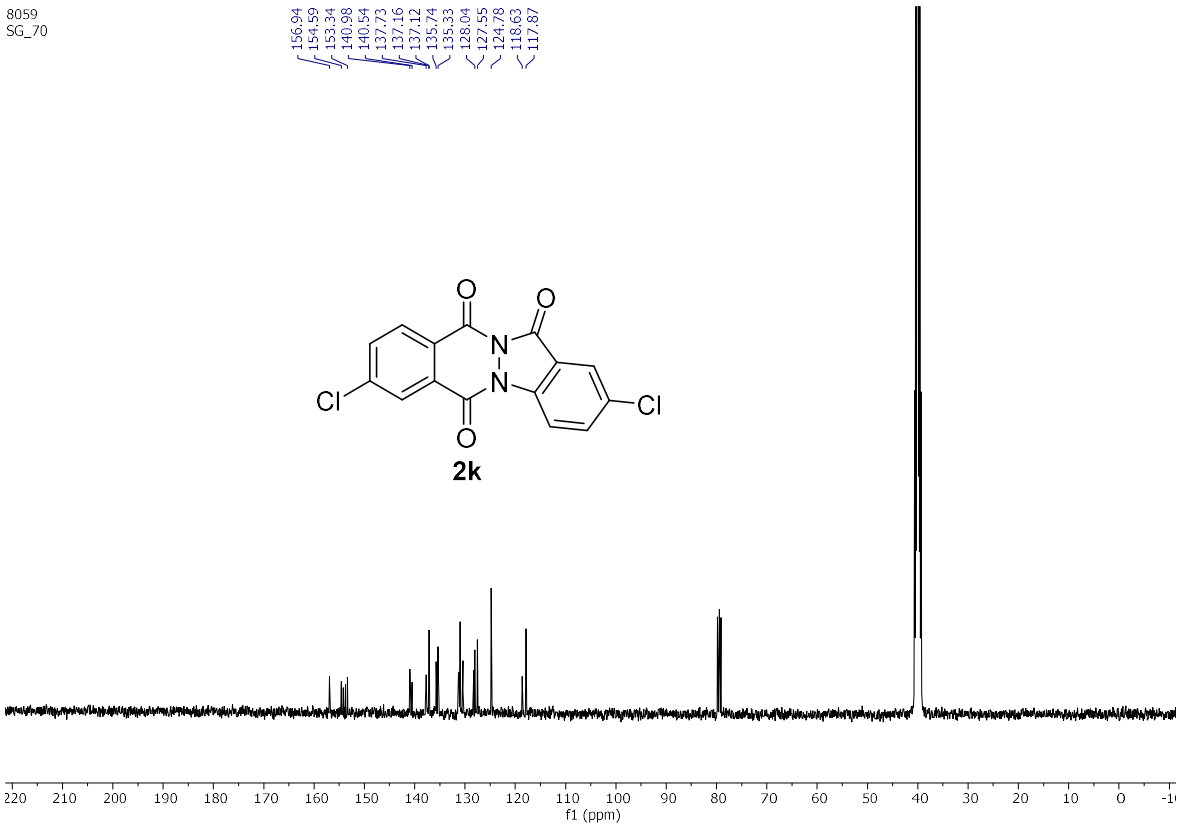
2j

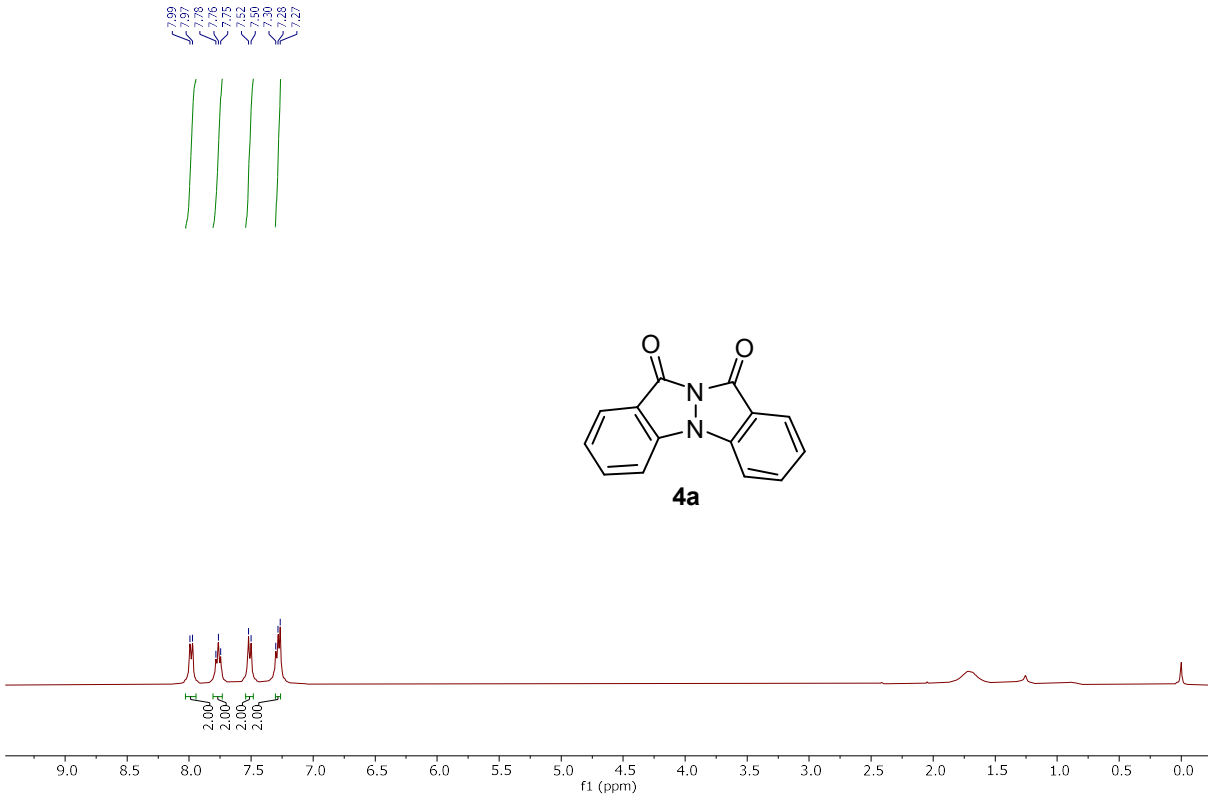


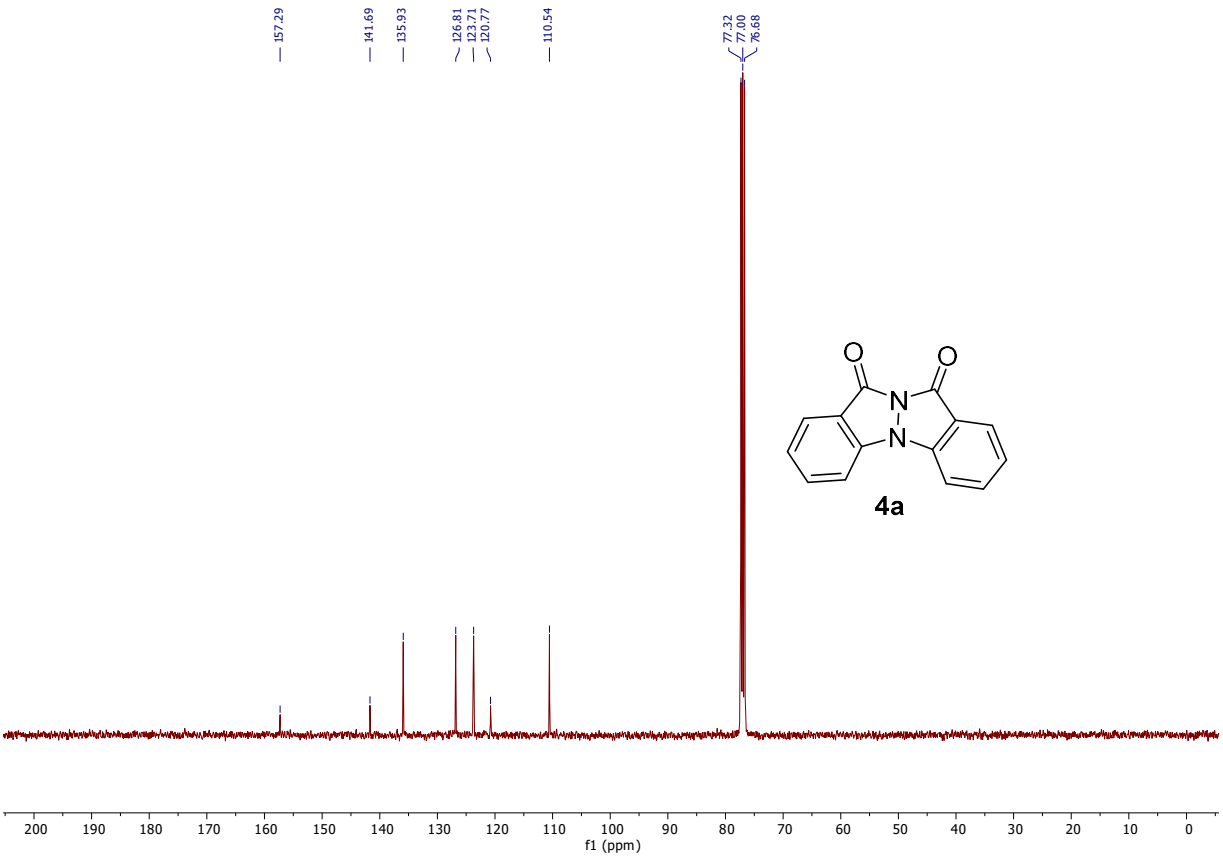


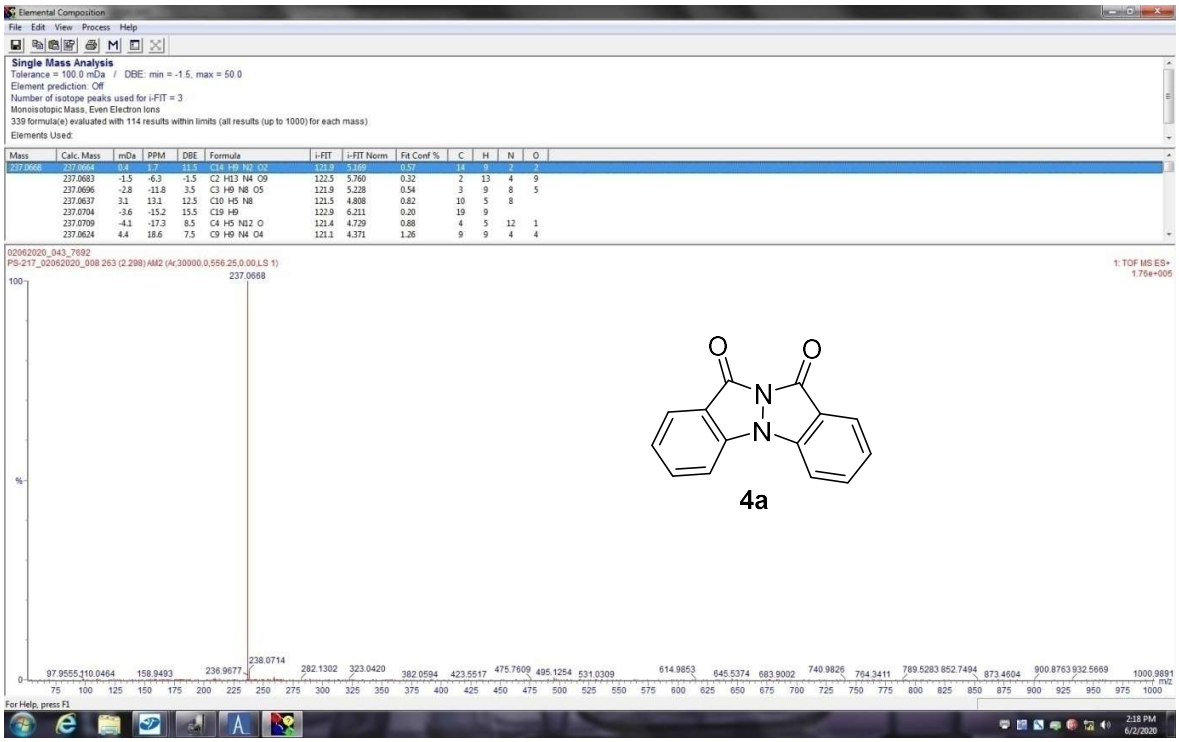
2k

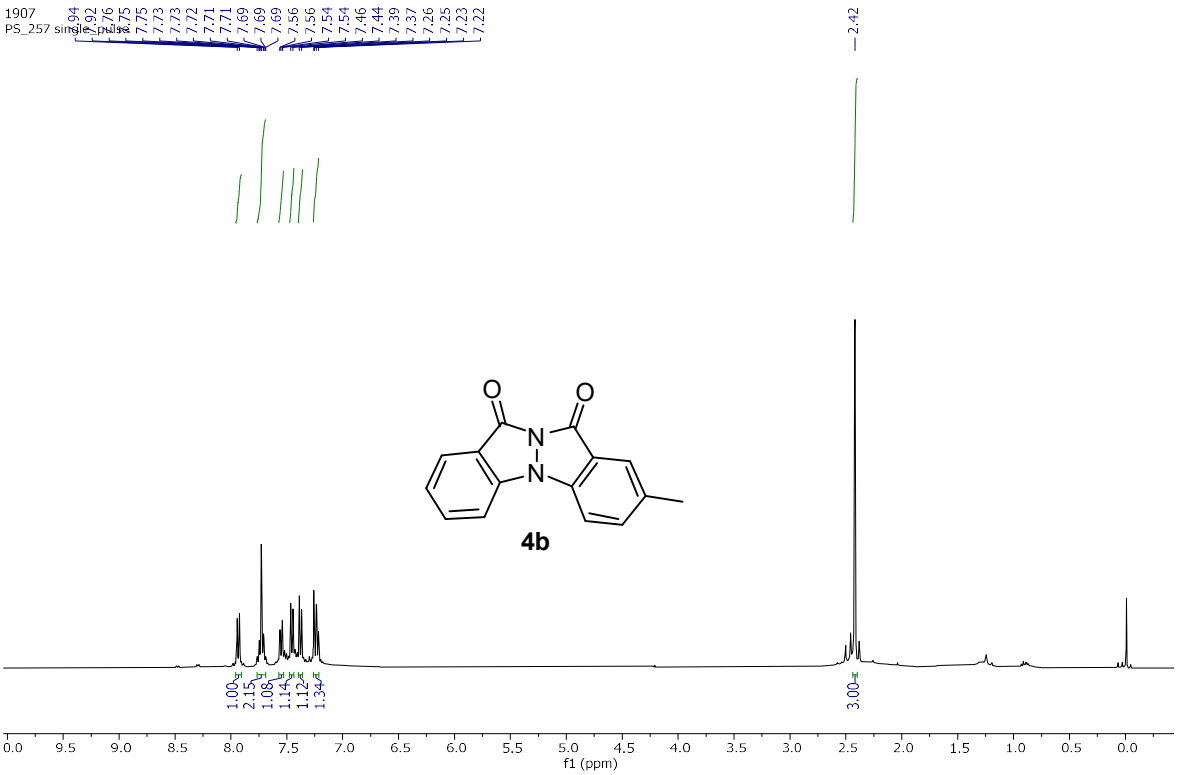

f1 (ppm)

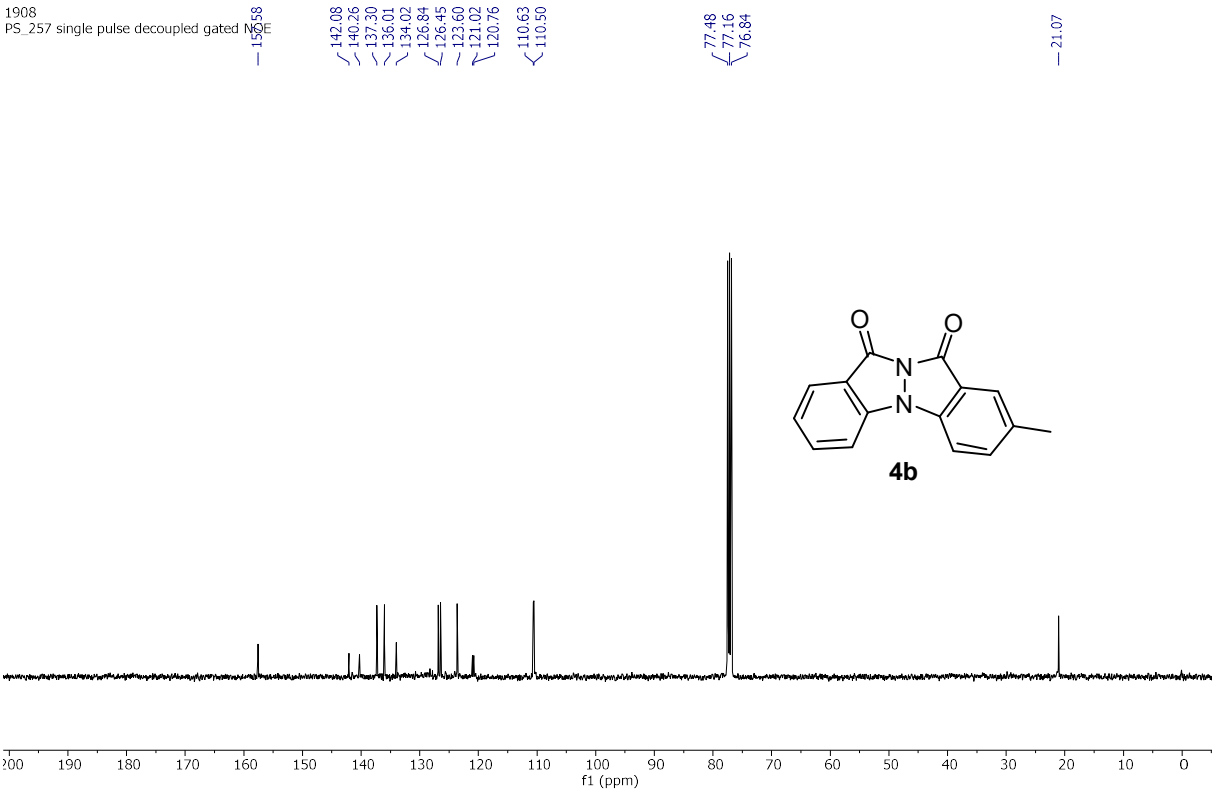

8059
SG_70
156.94
154.59
153.34
140.98
140.54
137.73
137.16
137.12
135.74
135.33
128.04
127.55
124.78
118.63
117.87
O
O
N
N
O
Cl
Cl
2k
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

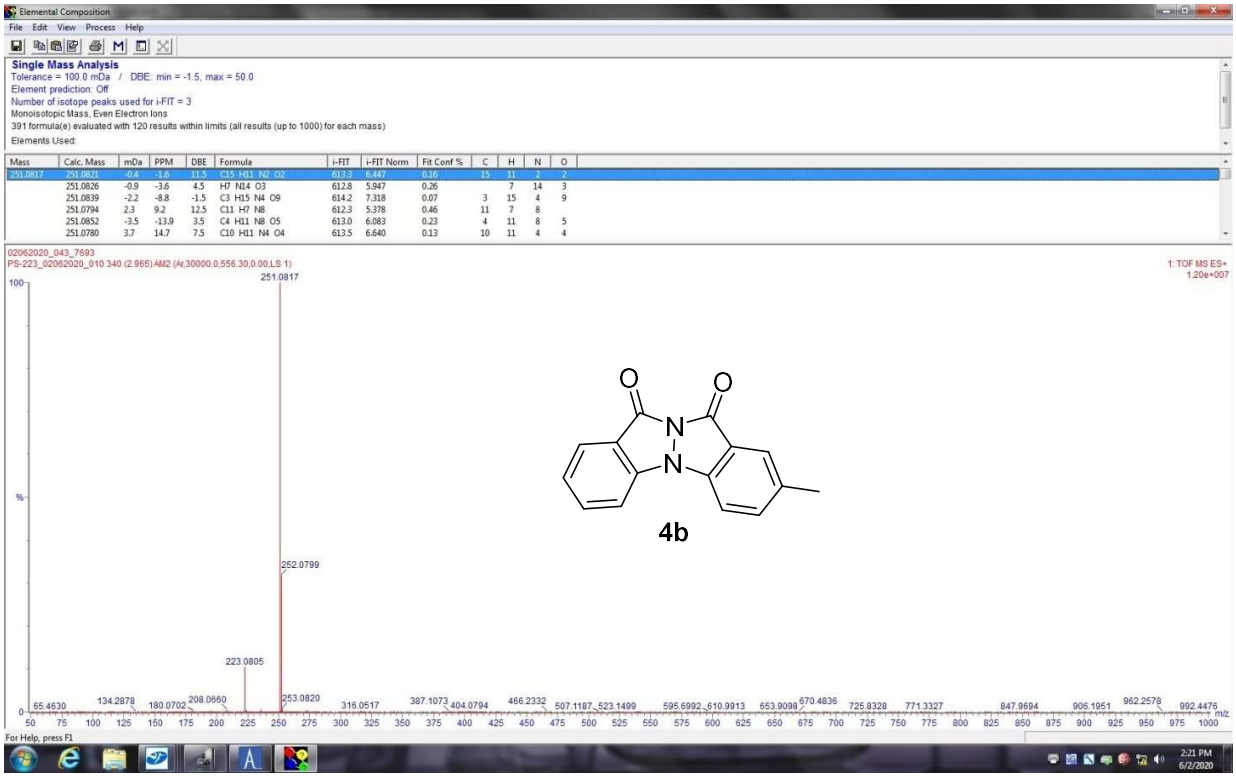

7.99
7.97
7.78
7.76
7.75
7.52
7.50
7.30
7.28
7.27
O
O
N
N
4a
2.00
2.00
2.00
2.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

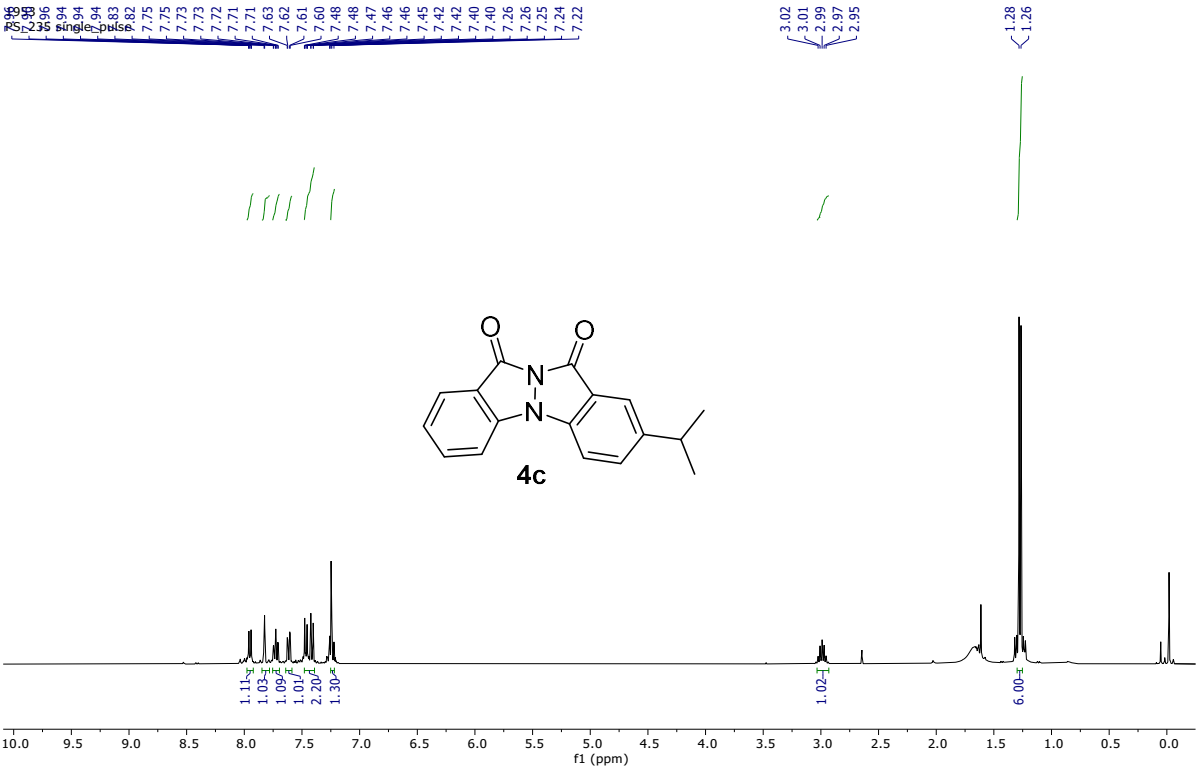

157.29
141.69
135.93
126.81
123.71
120.77
110.54
77.32
77.00
76.68
O
O
N
N
4a
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

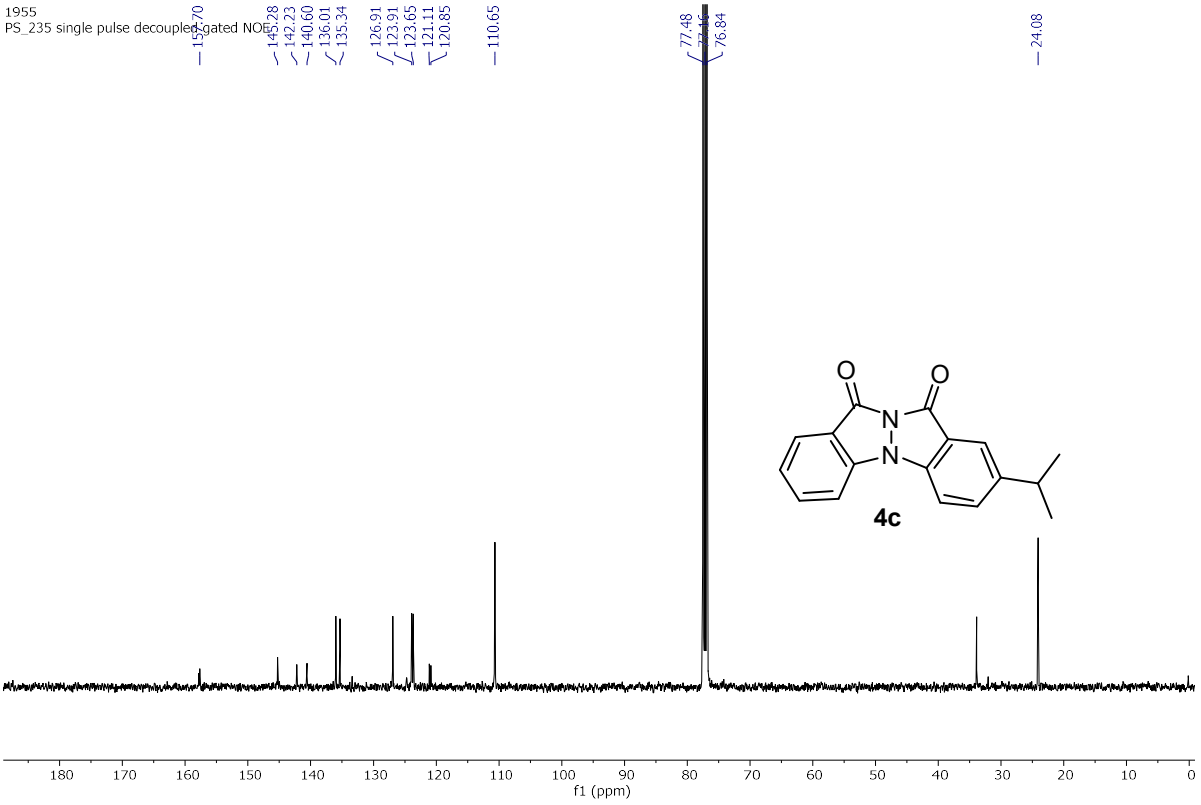

Elemental Composition
File Edit View Process Help
Single Mass Analysis
Tolerance = 100.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3
Monoisotopic Mass, Even Electron Ions
339 formula(e) evaluated with 114 results within limits (all results (up 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
237.0668 237.0664 0.4 1.7 11.5 C14 H9 N2 O2 121.9 5.169 0.52 14 9 2 2
237.0683 -1.5 -6.3 -1.5 C2 H13 N4 O9 122.5 5.760 0.32 2 13 4 9
237.0696 -2.8 -11.8 3.5 C3 H9 N8 O5 121.9 5.228 0.54 3 9 8 5
237.0637 3.1 13.1 12.5 C10 H5 N8 121.5 4.808 0.82 10 5 8
237.0704 -3.6 -15.2 15.5 C19 H9 122.9 6.211 0.20 19 9
237.0709 -4.1 -17.3 8.5 C4 H5 N12 O 121.4 4.729 0.88 4 5 12 1
237.0624 4.4 18.6 7.5 C9 H9 N4 O4 121.1 4.371 1.26 9 9 4 4
02062020_043_7692
PS-217_02062020_008 263 (2.298) AM2 (Ar,30000.0,556.25,0.00,LS 1)
1: TOF MS ES+
1.76e+005
237.0668
100
%
0
97.9555 110.0464 158.9493 236.9677 238.0714 282.1302 323.0420 382.0594 423.5517 475.7609 495.1254 531.0309 614.9863 645.5374 683.9002 740.9826 764.3411 789.5283 852.7494 873.4604 900.8763 932.5669 1000.9891
75 100 125 150 175 200 225 250 275 300 325 350 375 400 425 450 475 500 525 550 575 600 625 650 675 700 725 750 775 800 825 850 875 900 925 950 975 1000
m/z
For Help, press F1
O
O
N
N
4a

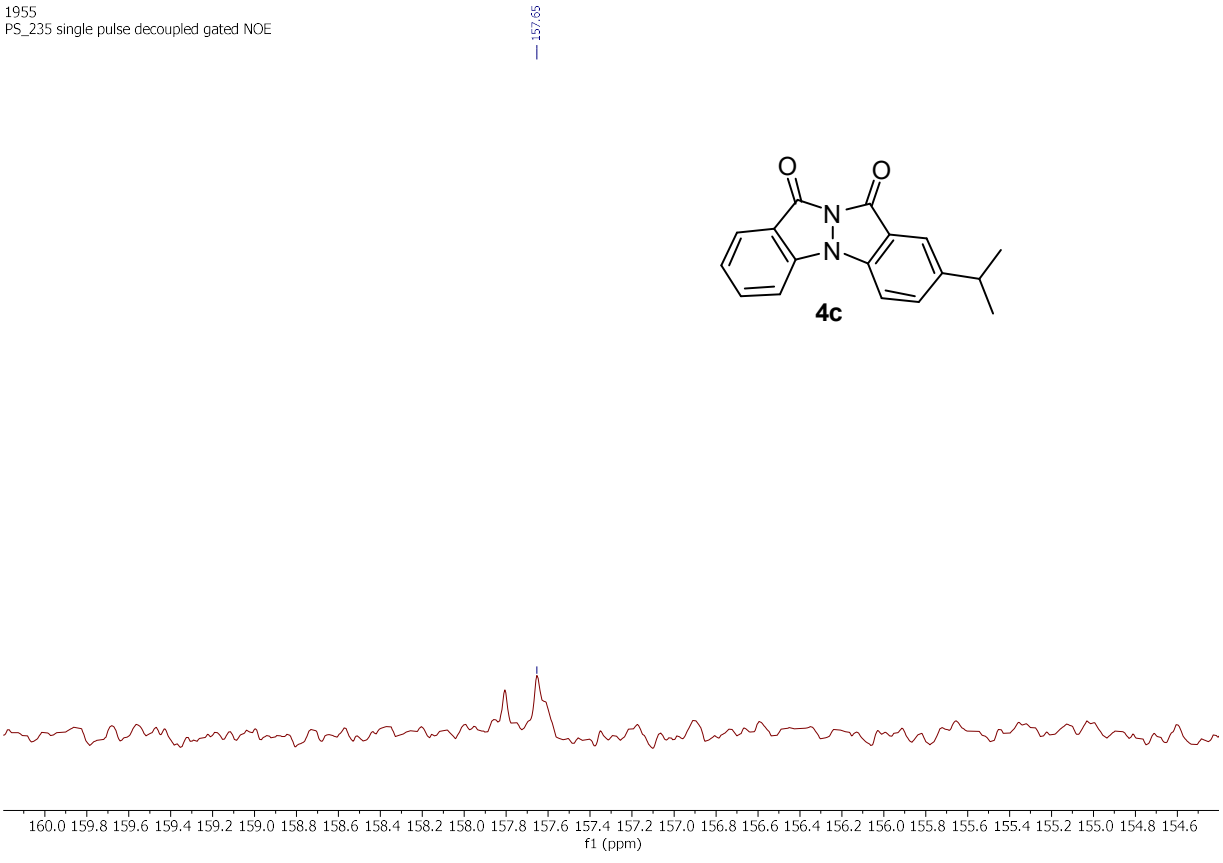

1907
PS_257 single pulse
7.94
7.92
7.76
7.75
7.75
7.73
7.73
7.72
7.71
7.71
7.69
7.69
7.69
7.56
7.56
7.54
7.54
7.46
7.44
7.39
7.37
7.26
7.25
7.23
7.22
2.42
O
O
N
N
4b
1.00
2.15
1.08
1.14
1.12
1.34
3.00
f1 (ppm)

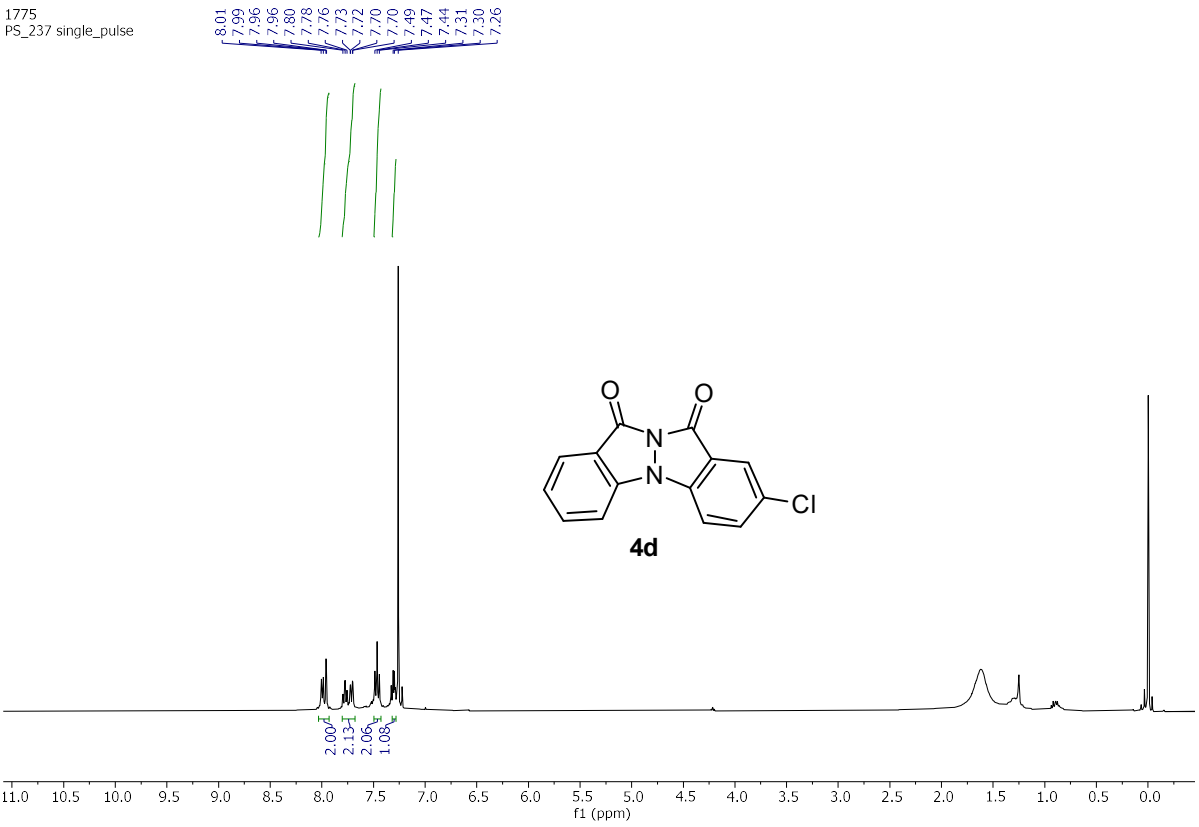

1908
PS_257 single pulse decoupled gated NOE
152.58
142.08
140.26
137.30
136.01
134.02
126.84
126.45
123.60
121.02
120.76
110.63
110.50
77.48
77.16
76.84
21.07
O
O
N
N
4b
f1 (ppm)

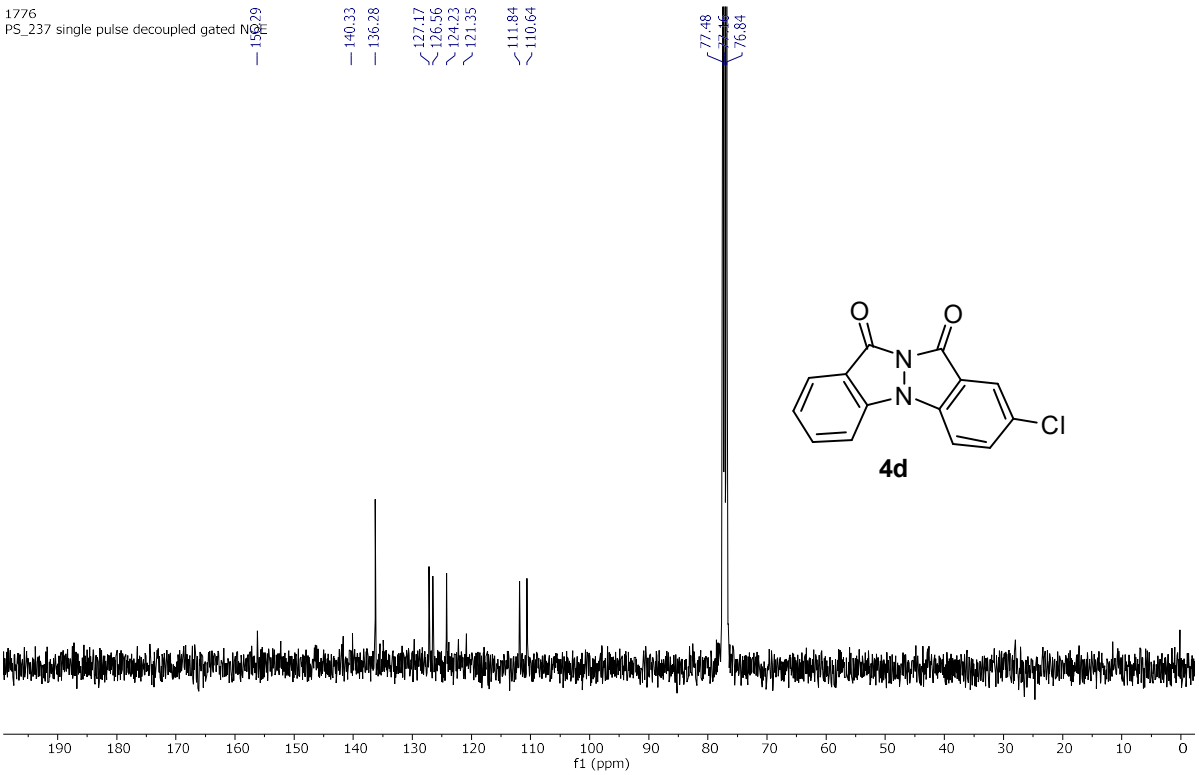

Elemental Composition
Single Mass Analysis
Tolerance = 100.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3
Monoisotopic Mass, Even Electron Ions
391 formula(e) evaluated with 120 results within limits (all results (up to 1000) for each mass)
Elements Used:
Mass Calc. Mass mDa PPM DBE Formula i-FIT i-FIT Norm Fit Conf % C H N O
251.0817 251.0821 -0.4 -1.6 11.5 C15 H11 N2 O2 613.3 6.447 0.16 15 11 2 2
251.0826 -0.9 -3.6 4.5 H7 N14 O3 612.8 5.947 0.26 7 14 3
251.0839 -2.2 -8.8 -1.5 C3 H15 N4 O9 614.2 7.318 0.07 3 15 4 9
251.0794 2.3 9.2 12.5 C13 H7 N8 612.3 5.378 0.46 11 7 8
251.0852 -3.5 -13.9 3.5 C4 H11 N8 O5 613.0 6.083 0.23 4 11 8 5
251.0780 3.7 14.7 7.5 C10 H11 N4 O4 613.5 6.640 0.13 10 11 4 4
02062020_043_7693
PS-223_02062020_010 340 (2.965) AM2 (Ar,30000.0,556.30,0.00,LS 1)
1: TOF MS ES+
1.20e+007
251.0817
252.0799
223.0805
4b

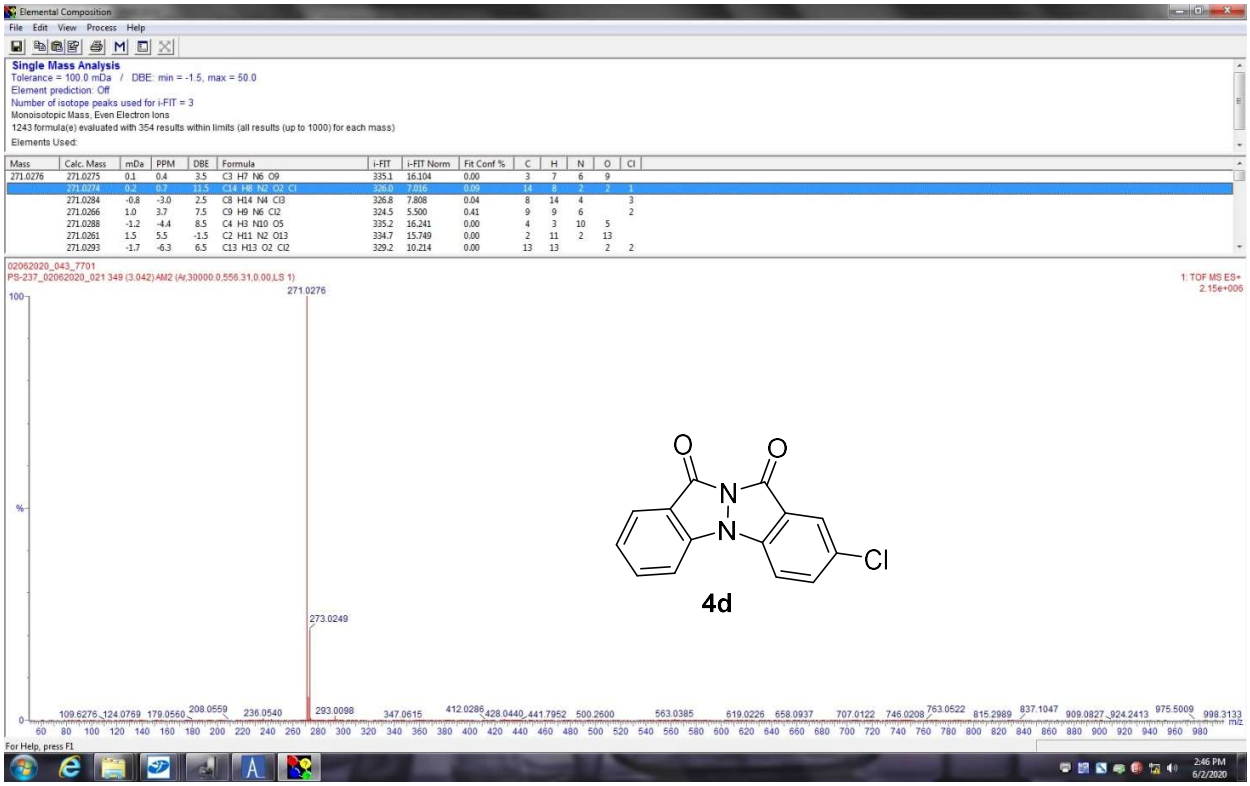

RS_235 single_pulse
4c
f1 (ppm)


1955
PS_235 single pulse decoupled gated NOE
152.70
145.28
142.23
140.60
136.01
135.34
126.91
123.91
123.65
121.11
120.85
110.65
77.48
77.16
76.84
24.08
O
O
N
N
4c
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


1955
PS_235 single pulse decoupled gated NOE
157.65
O
O
N
N
4c
160.0 159.8 159.6 159.4 159.2 159.0 158.8 158.6 158.4 158.2 158.0 157.8 157.6 157.4 157.2 157.0 156.8 156.6 156.4 156.2 156.0 155.8 155.6 155.4 155.2 155.0 154.8 154.6
f1 (ppm)

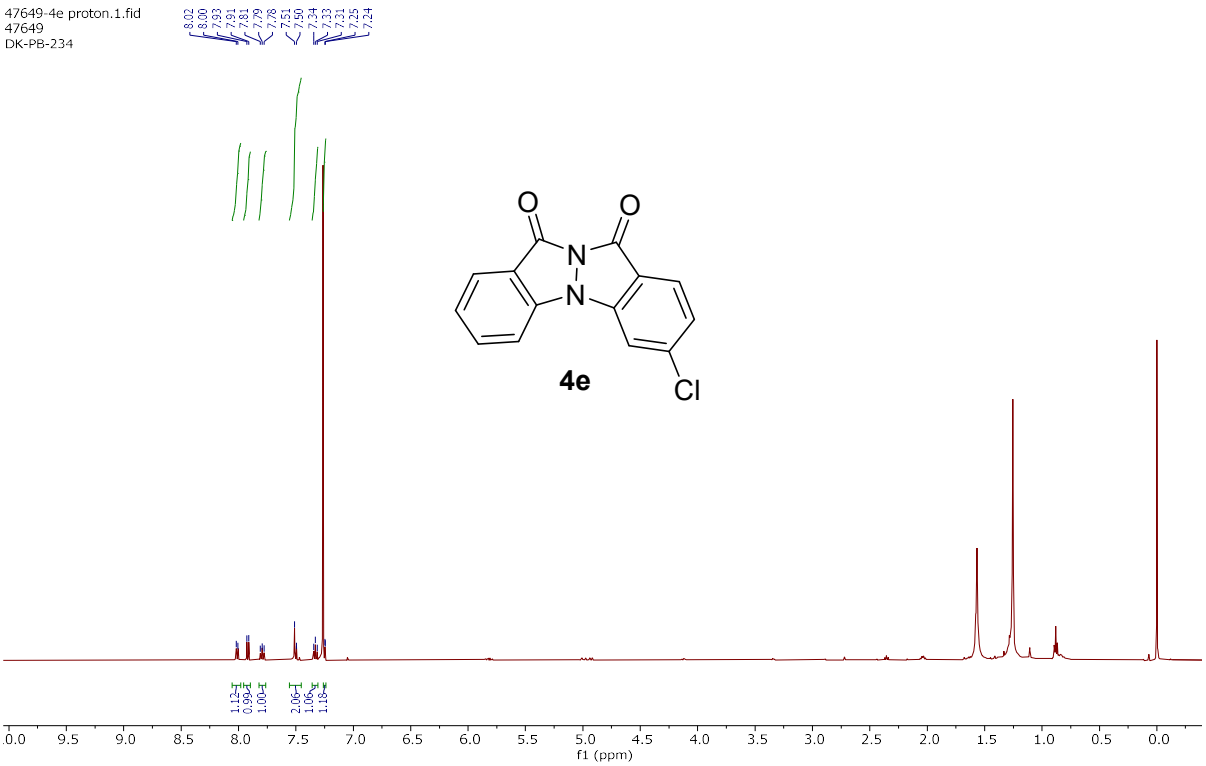

1775
PS_237 single_pulse
8.01
7.99
7.96
7.96
7.80
7.78
7.76
7.73
7.72
7.70
7.70
7.49
7.47
7.44
7.31
7.30
7.26
2.00
2.13
2.06
1.08
O
O
N
N
Cl
4d
f1 (ppm)

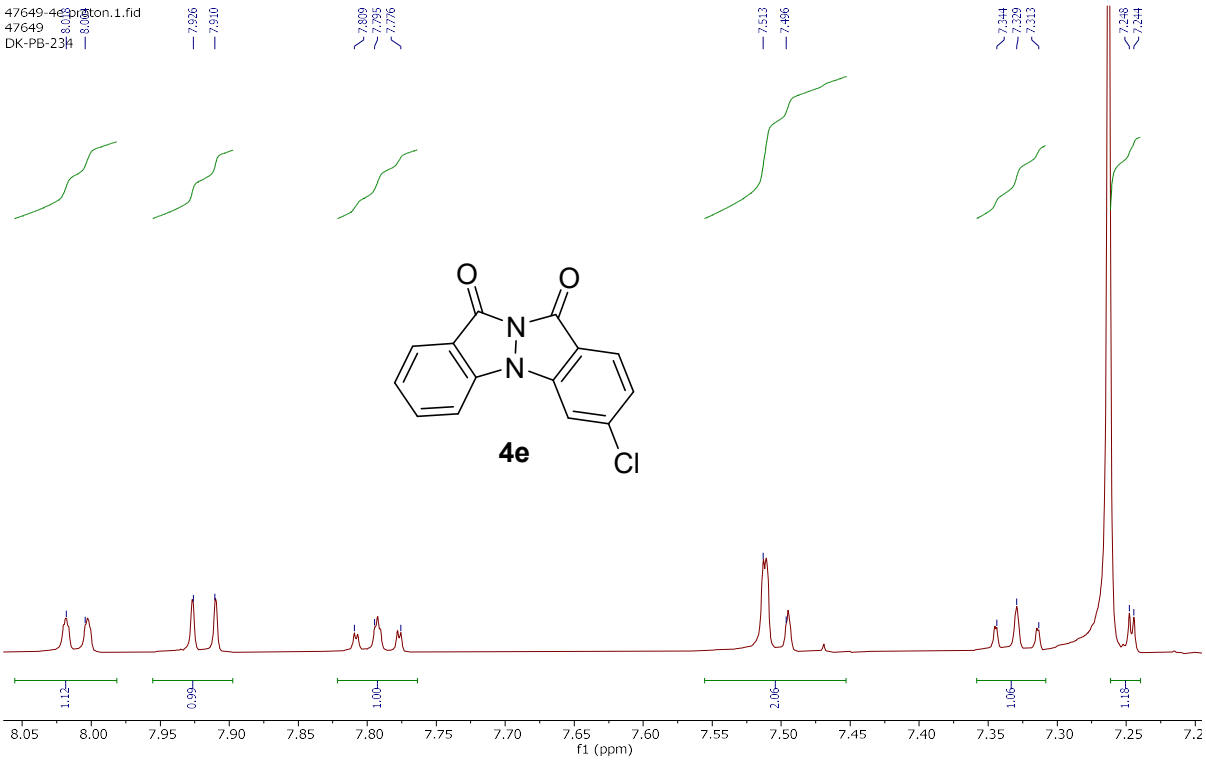

1776
PS_237 single pulse decoupled gated NOE
159.29
140.33
136.28
127.17
126.56
124.23
121.35
111.84
110.64
77.48
77.16
76.84
O
O
N
N
Cl
4d
f1 (ppm)

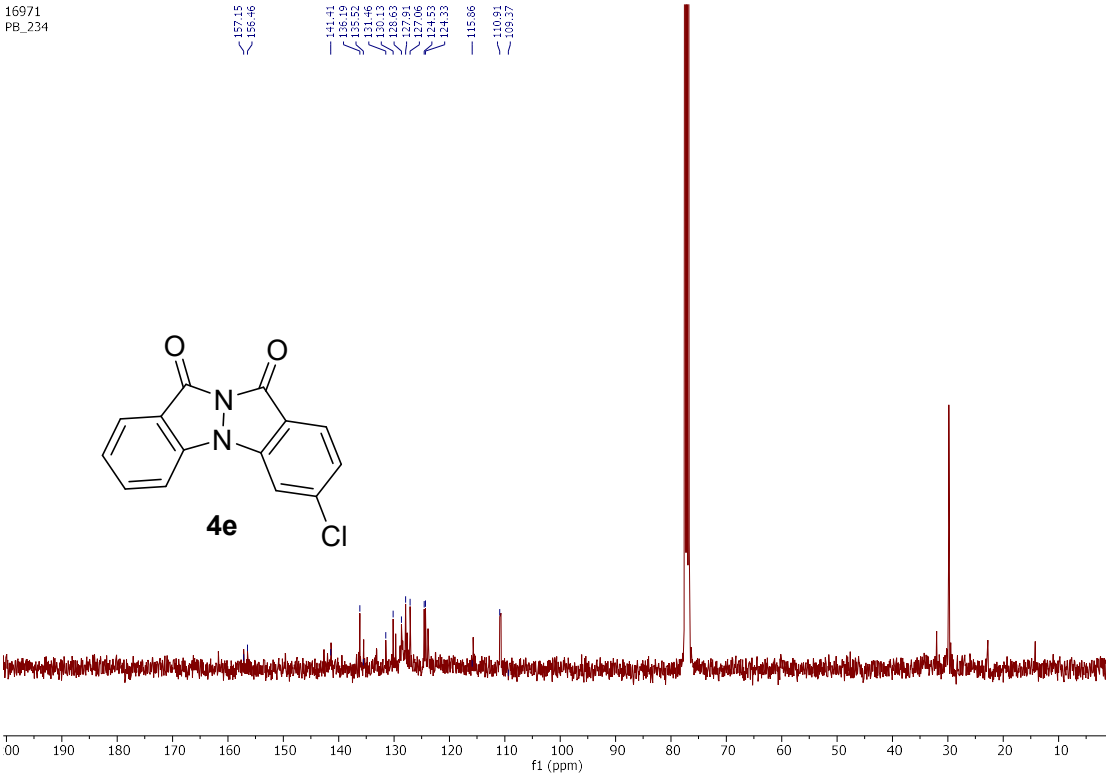

Elemental Composition
File Edit View Process Help
Single Mass Analysis
Tolerance = 100.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3
Monoisotopic Mass, Even Electron Ions
1243 formula(e) evaluated with 354 results within limits (all results (up 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
271.0276 271.0275 0.1 0.4 3.5 C3 H7 N6 O9 335.1 16.104 0.00 3 7 6 9
271.0274 0.2 0.7 11.5 C14 H8 N2 O2 Cl 326.0 7.016 0.09 14 8 2 2 1
271.0284 -0.8 -3.0 2.5 C8 H14 N4 Cl3 326.8 7.808 0.04 8 14 4 3
271.0266 1.0 3.7 7.5 C9 H9 N6 Cl2 324.5 5.500 0.41 9 9 6 2
271.0288 -1.2 -4.4 8.5 C4 H3 N10 O5 335.2 16.241 0.00 4 3 10 5
271.0261 1.5 5.5 -1.5 C2 H11 N2 O13 334.7 15.749 0.00 2 11 2 13
271.0293 -1.7 -6.3 6.5 C13 H13 O2 Cl2 329.2 10.214 0.00 13 13 2 2
02062020_043_7701
PS-237_02062020_021 349 (3.042) AM2 (Ar,30000.0,556.31,0.00,LS 1)
1: TOF MS ES+
2.15e+006
271.0276
273.0249
%
m/z
For Help, press F1
2:46 PM
6/2/2020

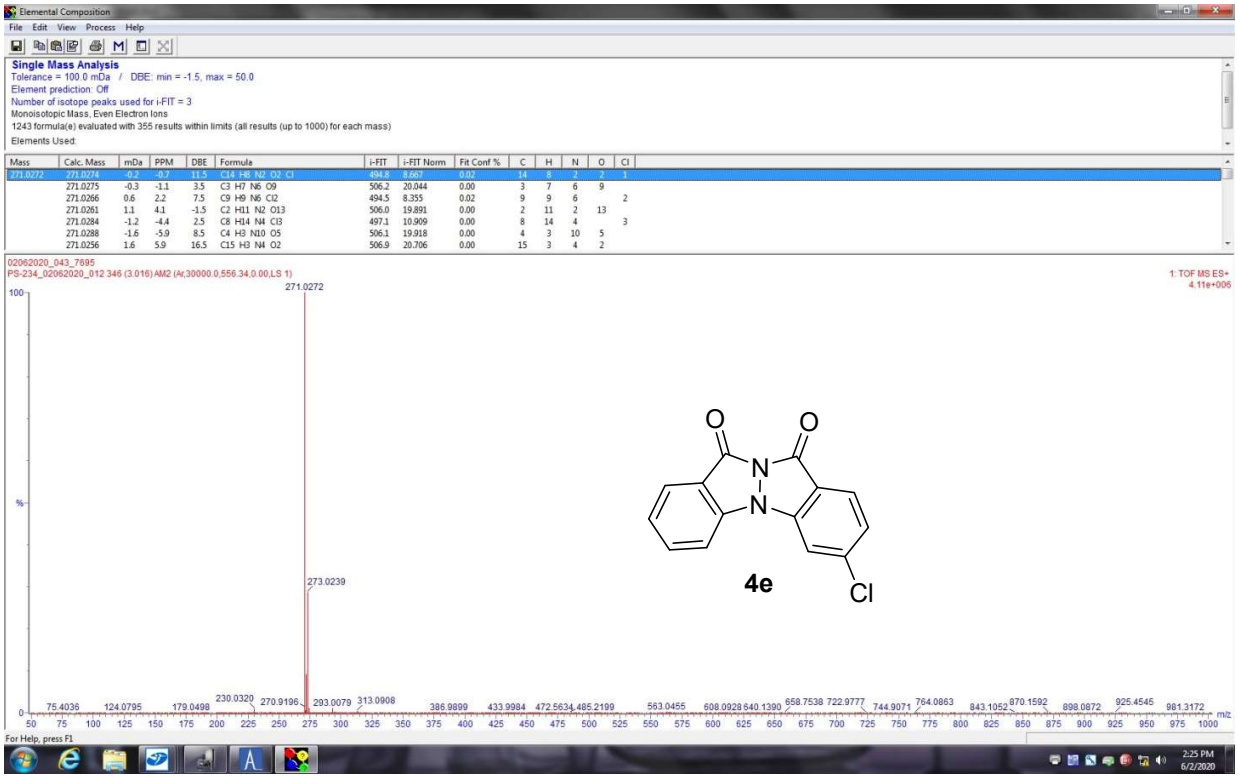
O
O
N
N
Cl

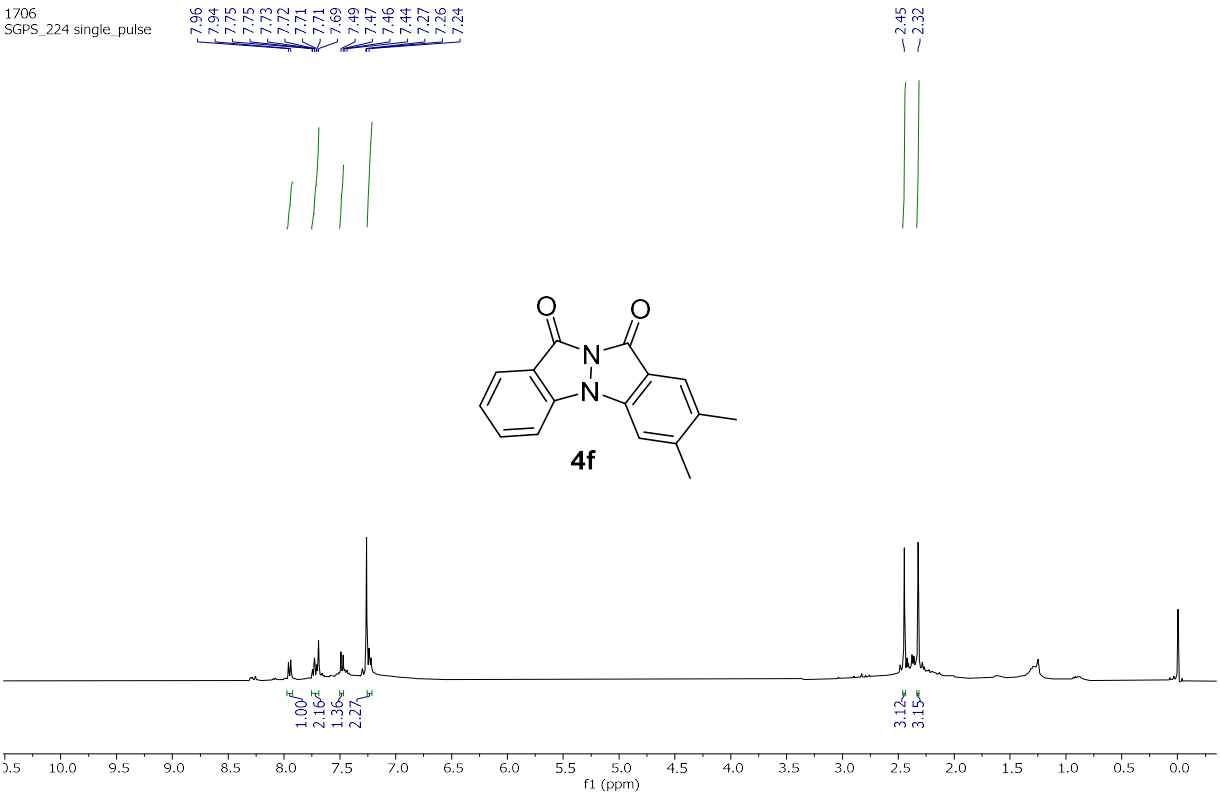
4d

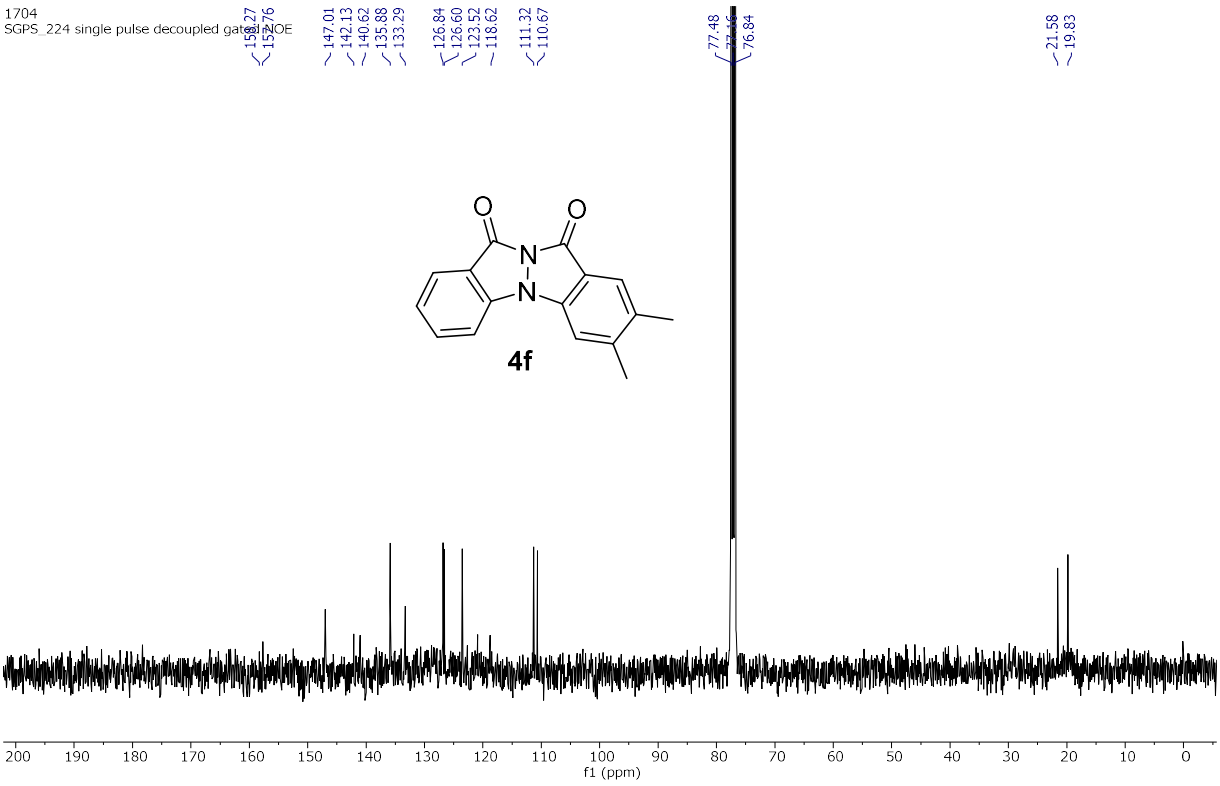

47649-4e proton.1.fid
47649
DK-PB-234
8.02
8.00
7.93
7.91
7.81
7.79
7.78
7.51
7.50
7.34
7.33
7.31
7.25
7.24
O
O
N
N
Cl
4e
1.12
0.99
1.00
2.06
1.06
1.18
f1 (ppm)

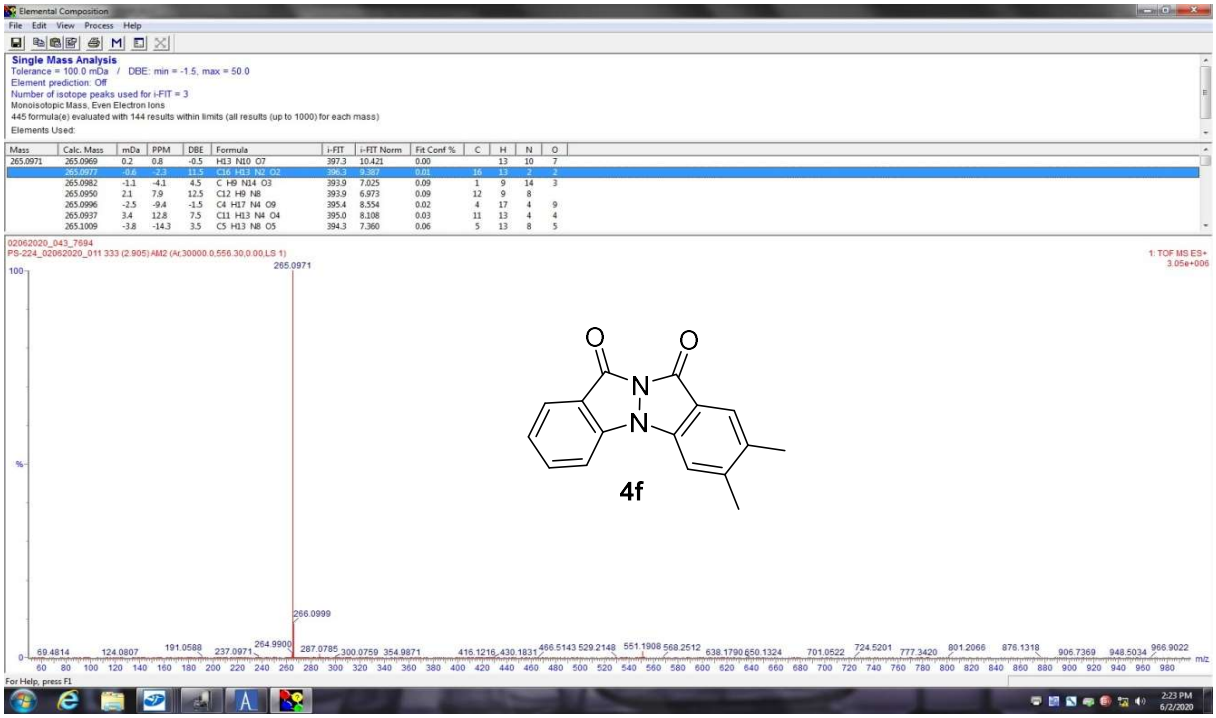

47649-4e proton.1.fid
47649
DK-PB-234
8.013
8.004
7.926
7.910
7.809
7.795
7.776
7.513
7.496
7.344
7.329
7.313
7.248
7.244
O
O
N
N
Cl
4e
1.12
0.99
1.00
2.06
1.06
1.18
f1 (ppm)

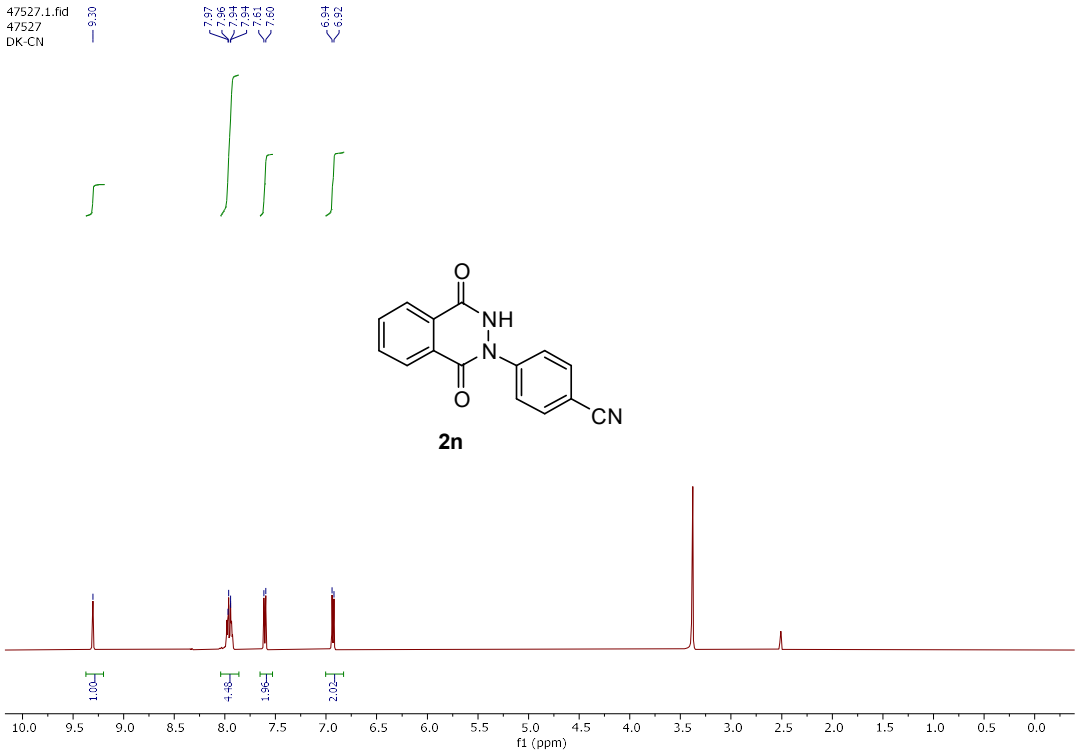

16971
PB_234
157.15
156.46
141.41
136.19
135.52
131.46
130.13
128.63
127.91
127.66
124.53
124.33
115.86
110.91
109.37
O
O
N
N
Cl
4e
f1 (ppm)


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
1243 formula(e) evaluated with 355 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
271.0272 271.0274 -0.2 -0.7 11.5 C14 H8 N2 O2 Cl 494.8 8.667 0.02 14 8 2 2 1
271.0275 -0.3 -1.1 3.5 C3 H7 N6 O9 506.2 20.044 0.00 3 7 6 9
271.0266 0.6 2.2 7.5 C9 H9 N6 Cl2 494.5 8.355 0.02 9 9 6 2
271.0261 1.1 4.1 -1.5 C2 H11 N2 O13 506.0 19.891 0.00 2 11 2 13
271.0284 -1.2 -4.4 2.5 C8 H14 N4 Cl3 497.1 10.909 0.00 8 14 4 3
271.0288 -1.6 -5.9 8.5 C4 H3 N10 O5 506.1 19.918 0.00 4 3 10 5
271.0256 1.6 5.9 16.5 C15 H3 N4 O2 506.9 20.706 0.00 15 3 4 2
02062020_043_7695
PS-234_02062020_012 346 (3.016) AM2 (Ar,30000.0,556.34,0.00,LS 1)
1: TOF MS ES+
4.11e+006
271.0272
273.0239
4e
Cl
For Help, press F1
2:25 PM
6/2/2020

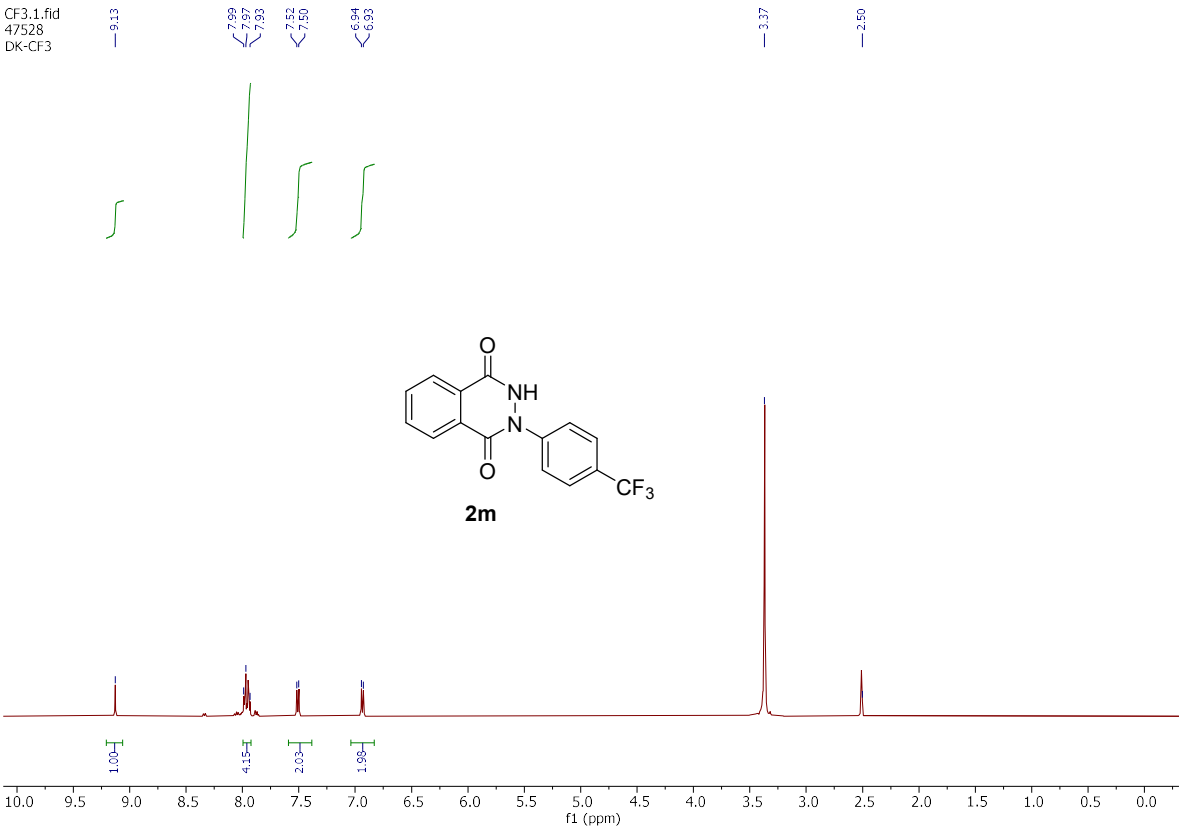

1706
SGPS_224 single_pulse
7.96
7.94
7.75
7.75
7.73
7.72
7.71
7.71
7.69
7.49
7.47
7.46
7.44
7.27
7.26
7.24
2.45
2.32
4f
1.00
2.16
1.36
2.27
3.12
3.15
f1 (ppm)


1704
SGPS_224 single pulse decoupled gated NOE
159.27
159.76
147.01
142.13
140.62
135.88
133.29
126.84
126.60
123.52
118.62
111.32
110.67
77.48
76.84
21.58
19.83
4f
f1 (ppm)

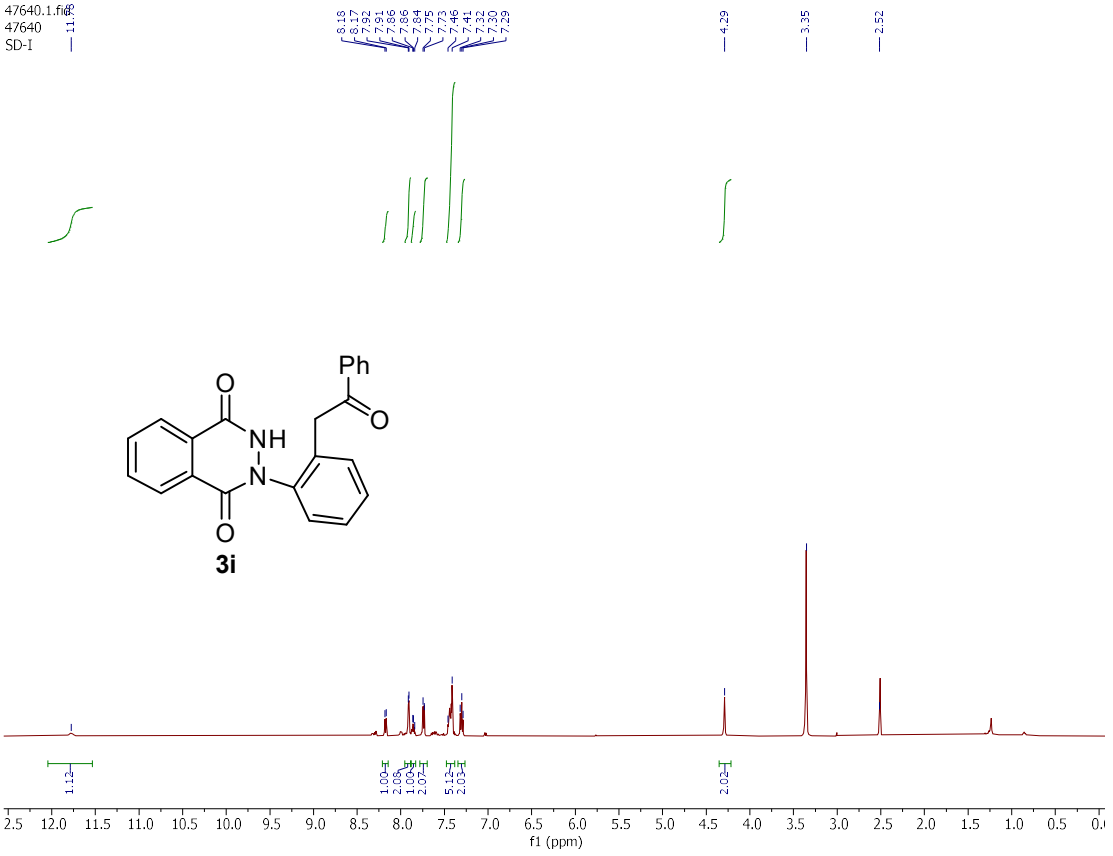

Elemental Composition
File Edit View Process Help
Single Mass Analysis
Tolerance = 100.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3
Monoisotopic Mass, Even Electron Ions
445 formula(e) evaluated with 144 results within limits (all results (up 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
265.0971 265.0969 0.2 0.8 -0.5 H13 N10 O7 397.3 10.421 0.00 13 10 7
265.0977 0.6 2.3 11.5 C16 H13 N2 O2 396.9 9.837 0.01 16 13 2
265.0982 1.1 4.1 4.5 C H9 N14 O3 393.9 7.025 0.09 1 9 14 3
265.0950 2.1 7.9 12.5 C12 H9 N8 393.9 6.973 0.09 12 9 8
265.0996 -2.5 -9.4 -1.5 C4 H17 N4 O9 395.4 8.554 0.02 4 17 4 9
265.0937 3.4 12.8 7.5 C11 H13 N4 O4 395.0 8.108 0.03 11 13 4 4
265.1009 -3.8 -14.3 3.5 C5 H13 N8 O5 394.3 7.360 0.06 5 13 8 5
02062020_043_7694
PS-224_02062020_011 333 (2.905) AM2 (Ar,30000.0,556.30,0.00,LS 1)
1: TOF MS ES+
3.05e+006
265.0971
266.0999
4f
For Help, press F1
2:23 PM
6/2/2020

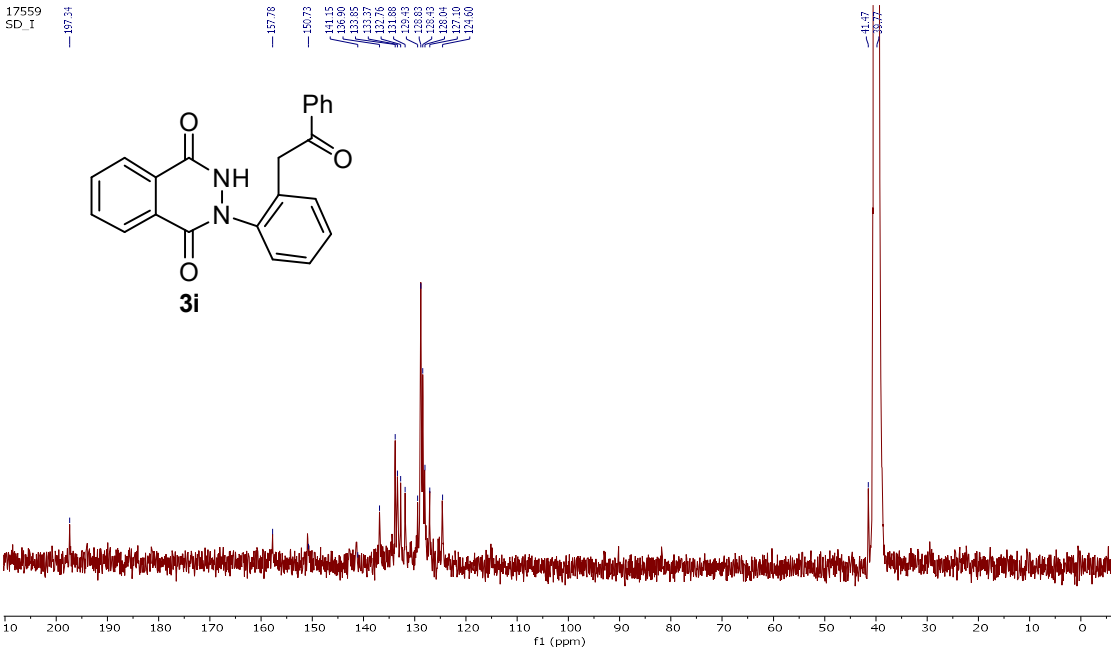

47527.1.fid
47527
DK-CN
9.30
7.97
7.96
7.94
7.94
7.61
7.60
6.94
6.92
O
NH
N
O
CN
1.00
4.48
1.96
2.03
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

2n


CF3.1.fid
47528
DK-CF3
9.13
7.99
7.97
7.93
7.52
7.50
6.94
6.93
3.37
2.50
O
NH
N
O
CF3
1.00
4.15
2.03
1.98
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)

2m


47640.1.fid
47640
SD-I
11.45
8.18
8.17
7.92
7.91
7.86
7.84
7.75
7.73
7.46
7.41
7.32
7.30
7.29
4.29
3.35
2.52
Ph
O
NH
N
O
O
3i
1.12
1.00
2.06
1.00
2.07
5.12
2.03
2.02
f1 (ppm)


17559
SD_I
167.34
157.78
150.73
141.15
136.90
133.85
133.37
132.76
131.88
129.43
128.83
128.43
128.04
127.10
124.60
41.47
38.72
Ph
O
NH
N
O
O
3i
f1 (ppm)