Supporting Information

Target-Activated Streptavidin-Biotin Controlled Binding Probe

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Materials and instruments

Chemicals and reagents were purchased from Sigma-Aldrich and TCI and used without further purification. All solvents (DMSO, DMF, acetonitrile, dichloromethane, hexane, ethyl acetate, and methanol) were purchased from Sigma-Aldrich and TCI and used without further treatment or distillation. PBS buffer (0.9 mM KCl, 2.67 mM KH_2PO_4, 138 mM NaCl, 8.1 mM Na_2HPO_4) was diluted 10-times from commercially available concentrates supplied by Amersco. Thin layer chromatography (TLC) was performed on TLC-aluminum sheets (Silica gel 60 F254, Merck). Flash column chromatography was performed with silica gel (230-400 mesh, Merck). Reverse-phase column chromatography was performed with Lichroprep RP-18 (40-63µm). HPLC analysis was performed with analytical column (XBridge BEH C18 Column, 130Å, 5 mm, 4.6 mm x 250 mm). Products were purified by semi-preparative column (VP 150/21 Nucleosil 300-5 C18, Macherey-Nagel). PMA, SIN-1, 4-Hydroxy-TEMPO, Uric acid, *L*-NAME, catalase, Apocynin and superoxide dismutase (from bovine erythrocytes) were purchased from Sigma-Aldrich. 4-(Hydroxymethyl)phenylboronic acid was purchased from Alfa Aesar, FeTMPyP was from Calbiotech, Streptavidin-Cy5, -Alexa488 and -Cy3 were from Jackson ImmunoResearch Inc.

^{1}H, and ^{13}C nuclear magnetic resonance (NMR) spectra were recorded either on Varian Unityinova-500 or Mercury-400 with ^{1}H chemical shifts (δ) reported in ppm relative to the solvent residual signals of d-chloroform (7.24 ppm), d-methanol (3.30 ppm), d-DMSO (2.49 ppm), D_2O (4.79 ppm). ^{13}C chemical shifts (δ) were reported in ppm relative to the solvent residual signals of d-chloroform (77.0 ppm), d-methanol (49.0 ppm) and d-DMSO (39.5 ppm). Coupling constants were reported in Hz. High resolution mass spectra (HRMS) were recorded on Varian 901-FTMS.

Preparation of various ROS and RNS solutions for selectivity studies

1. Nitrate (NO_3^-) and nitrite (NO_2^-) were prepared individually from sodium nitrate and sodium nitrite. They were dissolved in ddH_2O to make up 20 mM stock solution.

2. Hydroxyl radical was generated by the Fenton reaction. To prepare 10 mM $OH^\bullet$ stock solution, 0.01 M H_2O_2 was mixed with 3.92 mg $(NH_4)_2Fe(SO_4)_2$ in 1 mL ddH_2O.

3. Nitric oxide (NO) stock solution (2 mM) was prepared by bubbling NO into deoxygenated deionized water for 30 min.

4. 1 mM superoxide solution was prepared by dissolving KO_2 in DMSO.

5. Peroxynitrite was generated from SIN-1. SIN-1 was dissolved in ddH_2O to prepare 20 mM stock solution.

6. 10 mM solution of HOCl in ddH_2O was diluted from commercial NaOCl solution (5% available chlorine, J.T. Baker)

7. 10 mM solution of H_2O_2 in ddH_2O was diluted from commercial H_2O_2 solution (10 M, SHOWA)

Subculture of RAW264.7 macrophages

RAW264.7 macrophages were grown in DMEM medium supplemented with 10% FBS and 1% penicillin-streptomycin at 37 °C with 5% CO_2. The cells were passaged after they reach 80 % confluence. To detach the cells, a sterilized spatula was used to gently and slowly scratch the cells. 5 mL DMEM medium were added to the flask and the cells were transferred to a 15 mL Falcon tube for centrifugation. The cells were re-suspended in 1 mL pre-warmed complete DMEM medium and an appropriate volume (~ 100 μL) was transferred to a new flask for culturing.

Imaging of peroxynitrite at the cell surface of RAW264.7 macrophages

RAW264.7 cells were maintained in DMEM medium supplemented with 10% FBS and 1% penicillin-streptomycin. 10^5 cells were seeded in 8-well chamber slides (Thermo-Nunc) and cultured overnight at 37 °C with 5% CO_2. The cells were washed three times with Opti-MEM and the nucleuses were stained with 0.1 μM Hoechst 34580. After 30 minutes, excess Hoechst 34580 was removed. The cells were labeled with 10 μM **ONOO-CBP** in HBSS buffer (1.0 % ACN, v/v) for 30 minutes at 37 °C. For the detection of exogenous $ONOO^-$, corresponding concentrations of SIN-1 in DMEM were added to the cells and incubated for 1 hour. For the imaging of secreted $ONOO^-$, the cells were treated with 1 μg/mL PMA for the indicated period in DMEM medium. To image the cells, 2 μg streptavidin-Cy5 was added and incubated for 10 minutes. After removing the unbound streptavidin-Cy5, the images were taken using Laser Scanning Confocal Microscope (LSM 700, Zeiss, Germany). For streptavidin-Cy5, the images were taken by using 639 nm laser and LP640 emission filter. For streptavidin-Cy3, the images were taken by using 555 nm laser and SP640 emission filter. For streptavidin-Alexa488, the images were taken by using 488 nm laser and BP 490-555 emission filter. For Hoechst, we used 405 nm laser and SP490 emission filter.

Flow cytometry analysis

Raw 264.7 cells were cultured in 6 cm dish. After $ONOO^-$ stimulation by adding PMA, SOD, and streptavidin-Cy5, the cells were collected by scrapping and the aggregates were removed using a cell strainer (BD, #352340). The remaining cell suspensions were analyzed by flow cytometer (CytoFLEX, Beckman Coulter). An estimated 1×10^4 single cells were obtained from three treatments (control, PMA, PMA + SOD). The fluorescent intensity (APC, Cy5 channel) of each event was recorded and displayed by histogram overlay.

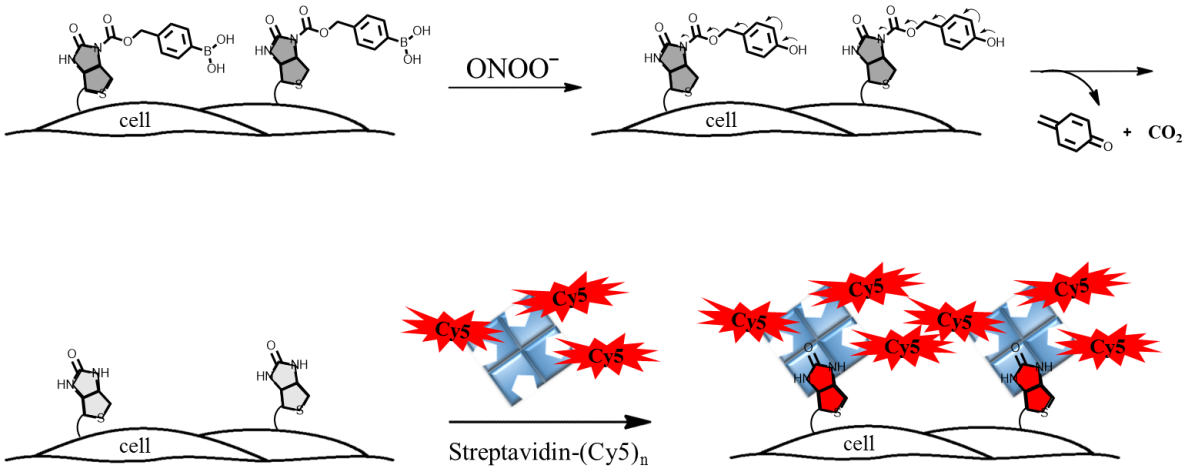


Fig. S1. Schematic illustration of membrane-anchored CBP-biotin probe **ONOO-CBP** for the imaging of $ONOO^-$ at the cell surface.

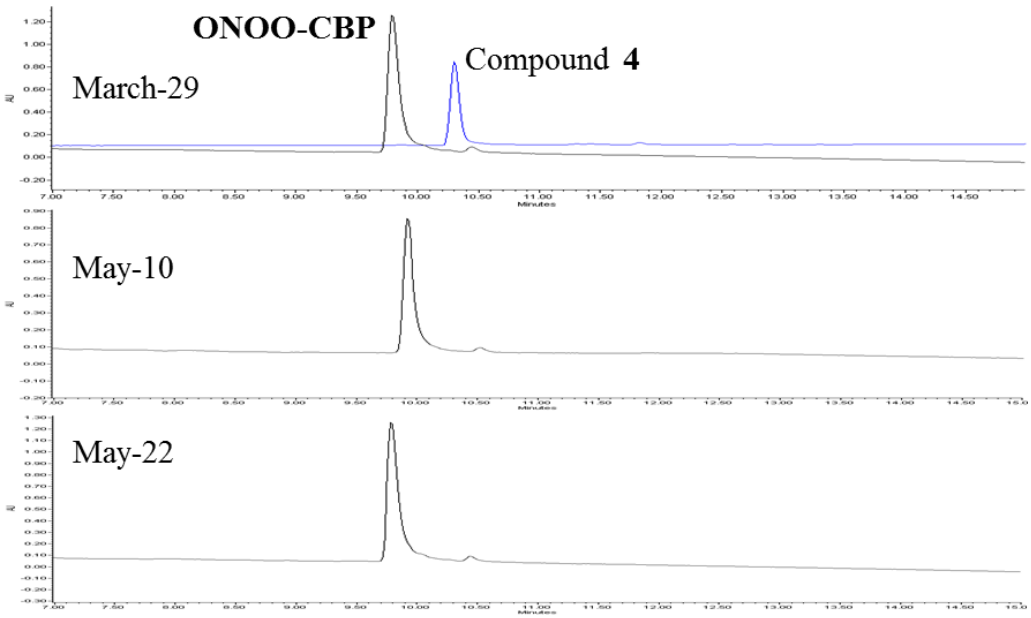


Fig. S2. Reversed-phase HPLC analysis of **ONOO-CBP** stored in an acidic medium at -80 °C. The HPLC traces show the same batch of **ONOO-CBP** analyzed on day 1 (29th March), day 43 (10th May) and day 55 (22nd May), respectively.

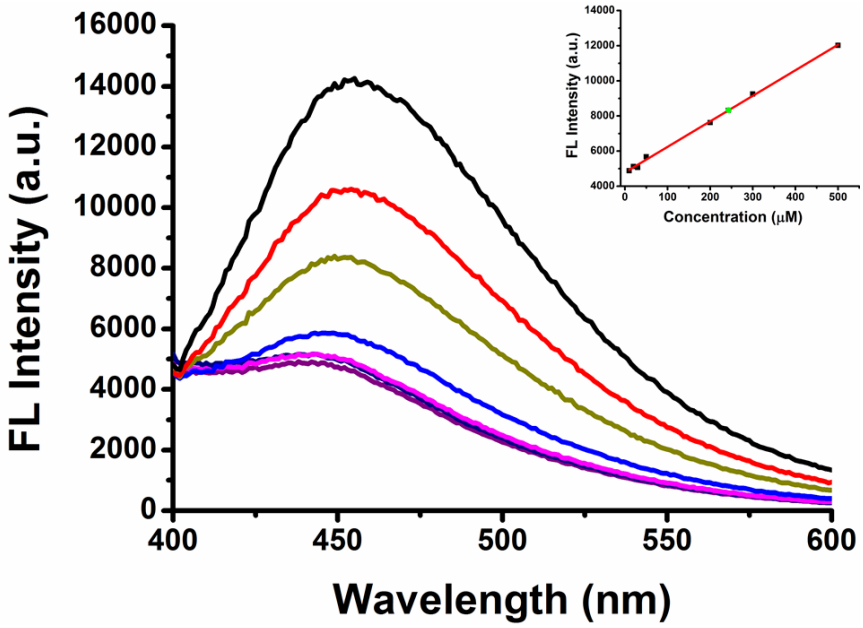


Fig. S3. Determination of the amount of $ONOO^-$ released from SIN-1 in one hour using nicotinamide adenine dinucleotide (reduced form, NADH). Detection principle: In the presence of $ONOO^-$, NADH can be oxidized to form weak fluorescence NAD^+. References: **1.** *J. Biol. Chem.*, 1999, **274**, 24664-24670. **2.** J. Fluoresc., 2004, **14**, 17-23. (a) Fluorescence spectra of different concentrations (10, 20, 30, 50, 200, 300 and 500 μM) of NADH in DMEM medium. Ex: 340 nm. The inset shows the fluorescence response of 250 μM NADH after one hour incubation with 100 μM SIN-1 (green dot). The amount of $ONOO^-$ released by 100 μM SIN-1 in one hour was determined to be around 7.33 ± 1.02 %. $y = 14.54x + 4785$, $R^2 = 0.99$.

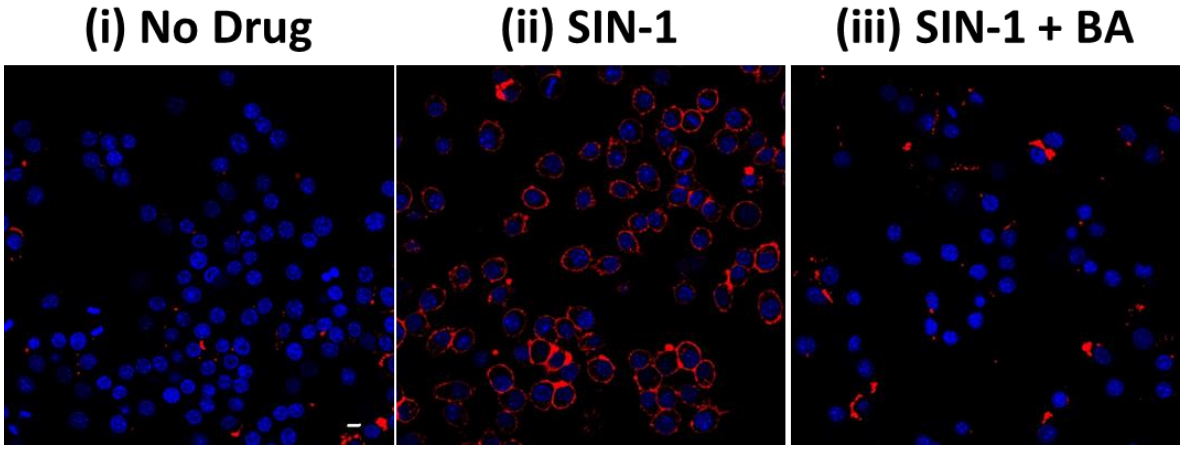


Fig. S4. Enlarged fluorescent images of **ONOO-CBP** labeled RAW264.7 cells in the (i) absence or (ii) presence of 100 μM SIN-1 or (iii) 250 μM BA with 100 μM SIN-1, after 1 hour incubation.

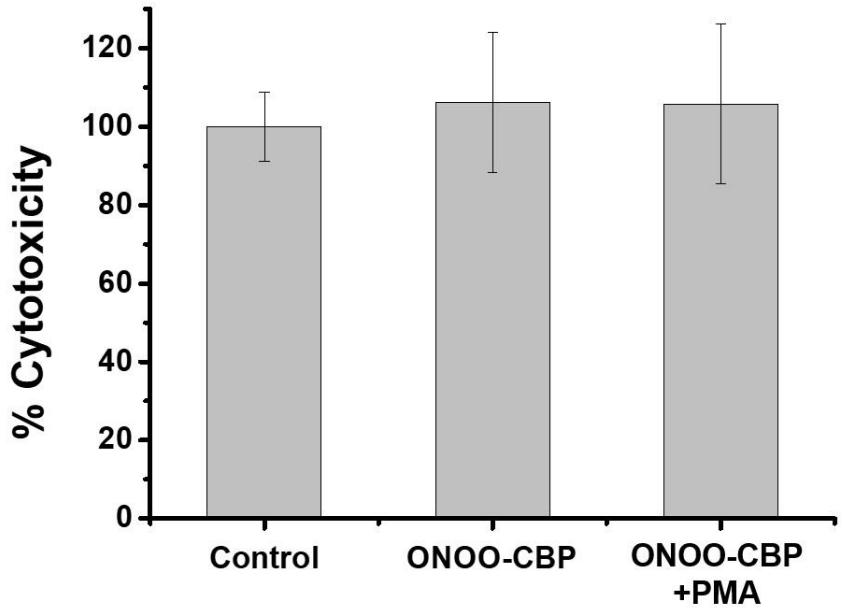


Figure S5. Cytotoxicity test of **ONOO-CBP** using MTT assay. 5 x 10^3 RAW264.7 cells were labeled with 10 μM **ONOO-CBP** for 30 minutes. Subsequently, the cells were treated with or without 1 μg/mL PMA for 1 hour in DMEM medium. Cytotoxicity test was conducted by following the standard MTT assay protocol (Vybrant® MTT Cell Proliferation Assay Kit, Thermo-Fisher Scientific). Control: Without **ONOO-CBP** labeling and PMA treatment.

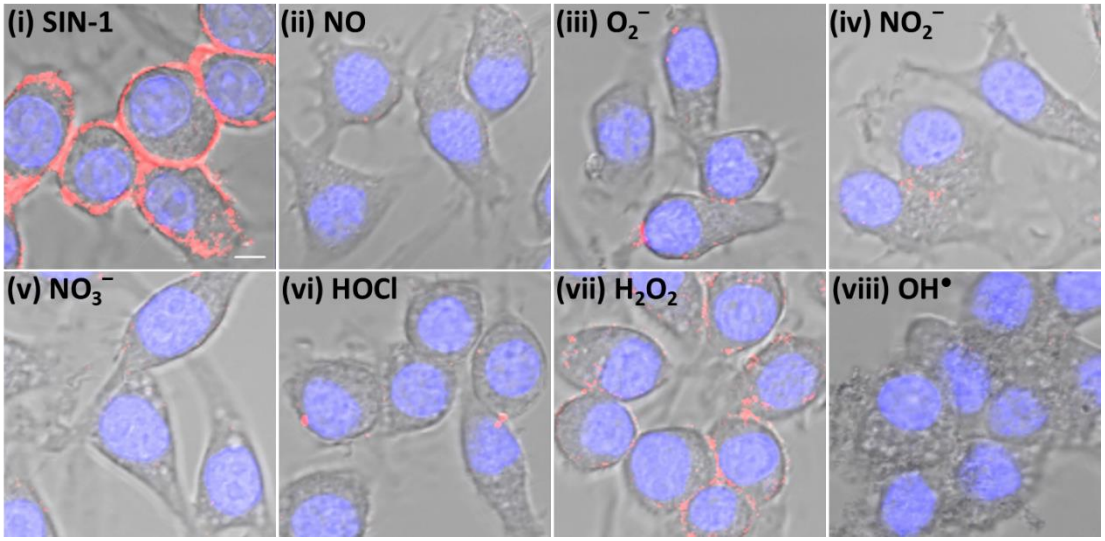
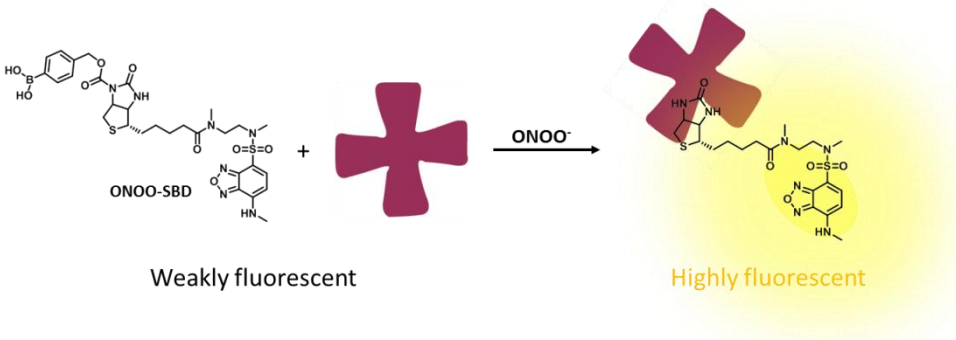


Fig. S6. Overlay of Cy5, Hoechst 34580 and bright field images of **ONOO-CBP** labeled RAW264.7 cells in the presence of (i) 50 μM SIN-1 or 100 μM each of (ii) NO, (iii) O_2^-, (iv) NO_2^-, (v) NO_3^-, (vi) HOCl, (vii) H_2O_2, (viii) $OH^•$. Cell surface stained with streptavidin-Cy5 are shown in red and the nuclei labeled with Hoechst 34580 are shown in blue. Scale bar: 20 μm.

(a)



(b)

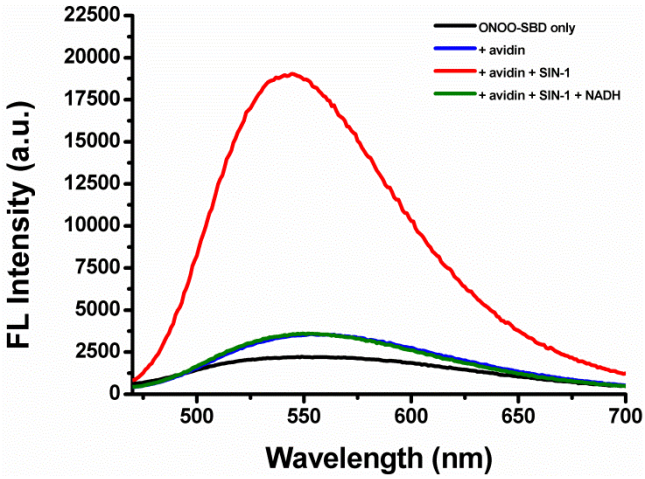
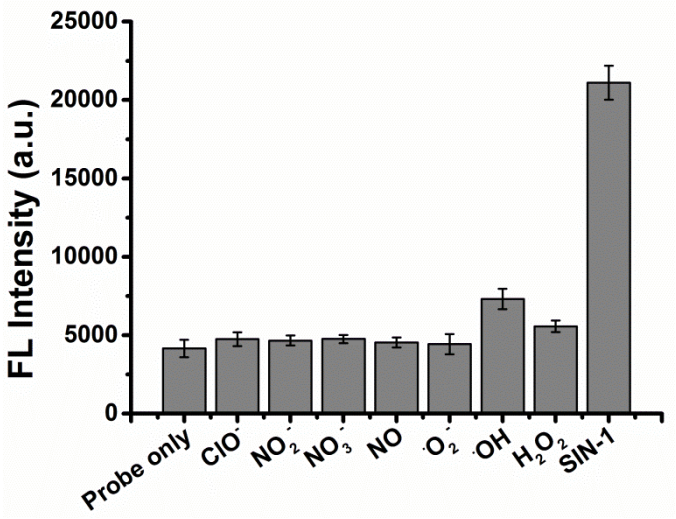


Fig. S7. (a) Schematic illustration of fluorogenic avidin-biotin controlled binding probe **ONOO-SBD** for the detection of $ONOO^-$ released from SIN-1 in the presence of avidin protein. (b) Fluorescence spectra of 20 μM **ONOO-SBD** in the absence (black line) or presence of 20 μM avidin (blue line), 100 μM SIN-1 (red line) or 250 μM NADH (green line). Ex : 440 nm.

(a)



(b)

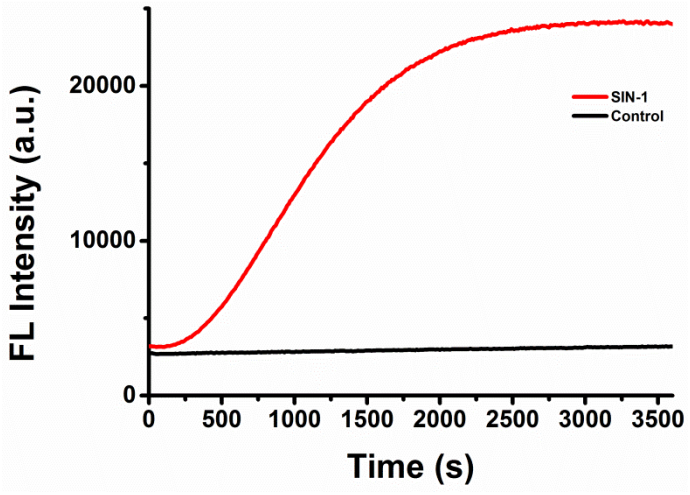
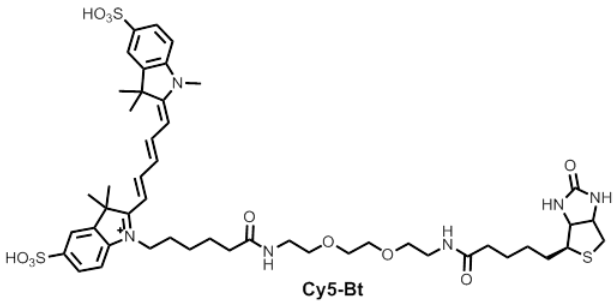
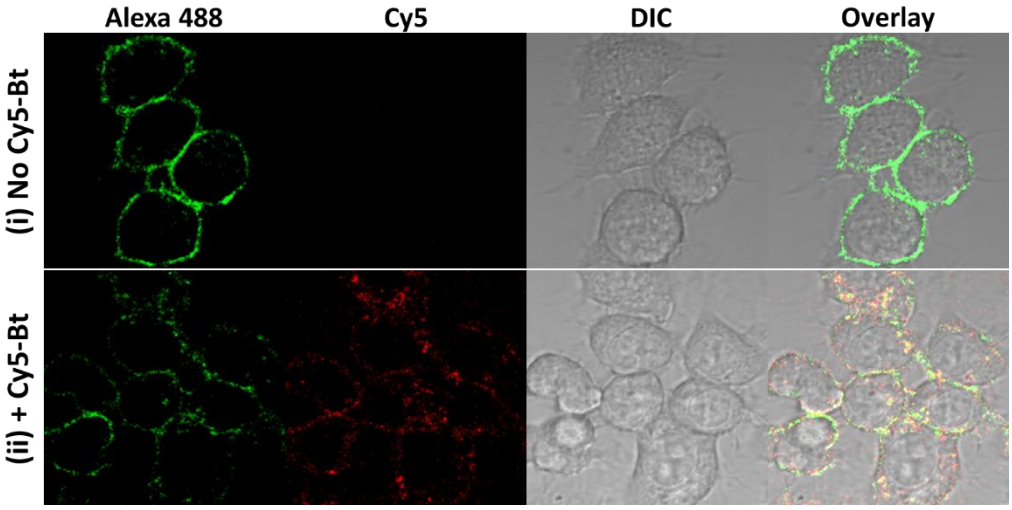


Fig. S8. (a) Selectivity test of 20 μM **ONOO-SBD** and 20 μM avidin mixture with 100 μM SIN-1 and seven non-targeted ROS/RNS (100 μM each). Error bars were calculated from triplicate measurements. (b) Fluorescence time course of 20 μM **ONOO-SBD** and 20 μM avidin mixture in the presence of 100 μM SIN-1.

(a)



(b)



(c)

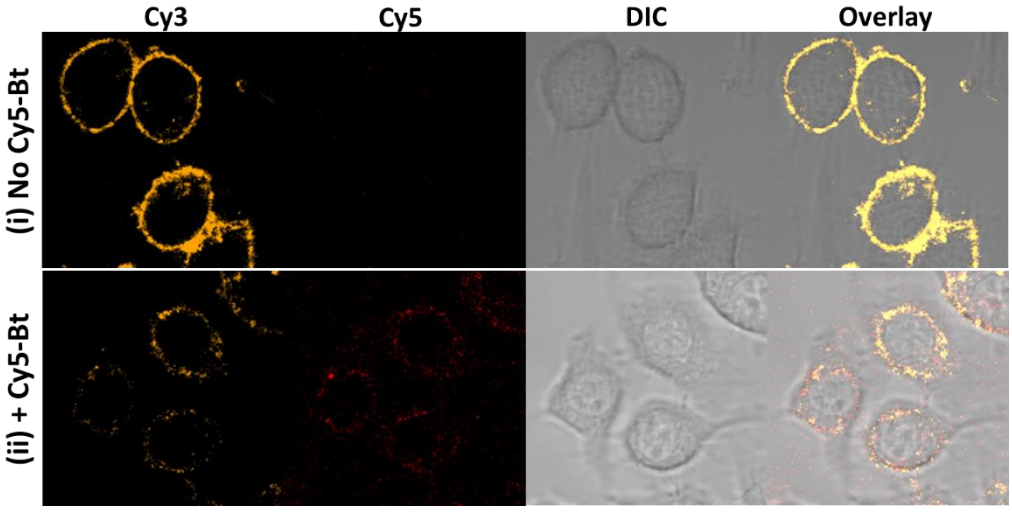


Fig. S9. (a) Chemical structure of **Cy5-Bt** for fluorescence amplification. Live cell imaging of **ONOO-CBP** labeled RAW264.7 cells in the presence of 50 μM SIN-1. (b) The cells were stained with (i) streptavidin-Alexa488 (ii) followed by the addition of **Cy5-Bt**. (c) The cells were stained with (i) streptavidin-Cy3 (ii) followed by the addition of **Cy5-Bt**.

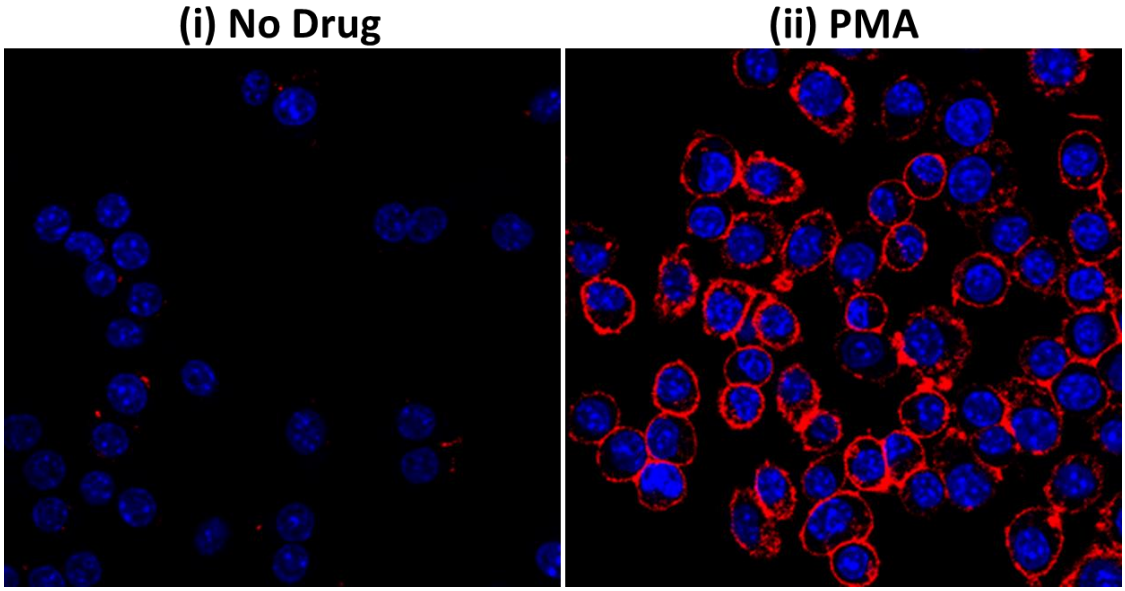


Fig. S10. Enlarged fluorescent images of **ONOO-CBP** labeled RAW264.7 cells in the (i) absence or (ii) presence of 1 μg/mg PMA after 1 hour incubation.

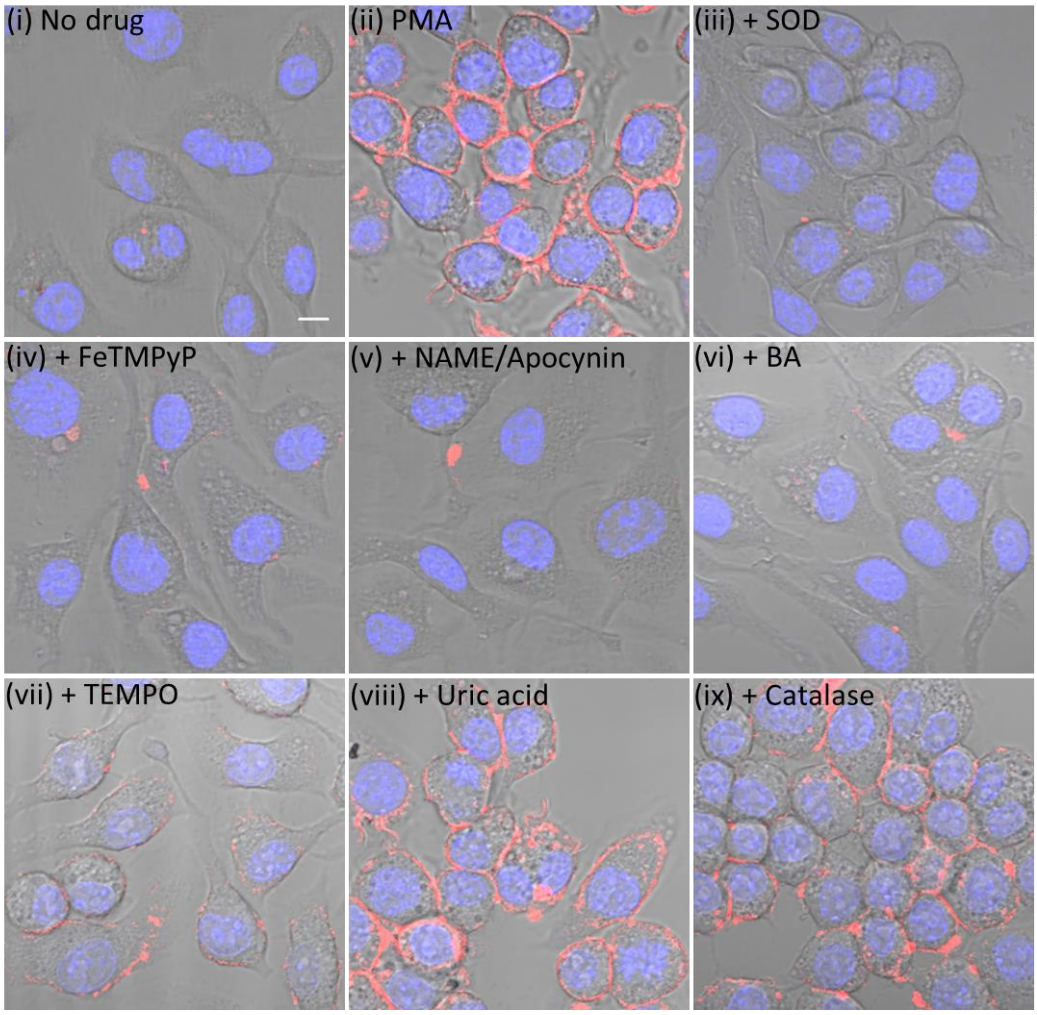


Fig. S11. Overlay of Cy5, Hoechst 34580 and bright field images of **ONOO-CBP** labeled RAW264.7 cells in in the (i) absence or (ii) presence of 1 μg/mL PMA and (iii) PMA + 15 μg/mL SOD, (iv) PMA + 1 mM FeTMPyP, (v) PMA + 1 mM L-NAME and 1 mM Apocynine, (vi) PMA + 250 μM BA, (vii) PMA + 1 mM TEMPO, (viii) PMA + 5 mM Uric acid, (ix) PMA + 10 μg/mL catalase. Besides L-NAME/Apocynin mixture, which were incubated with the cells for 90 minutes prior to PMA stimulation, all the inhibitors were added to the cells together with PMA and incubated for 60 minutes. The cells were stained with streptavidin-Cy5. Scale bar: 20 μm.

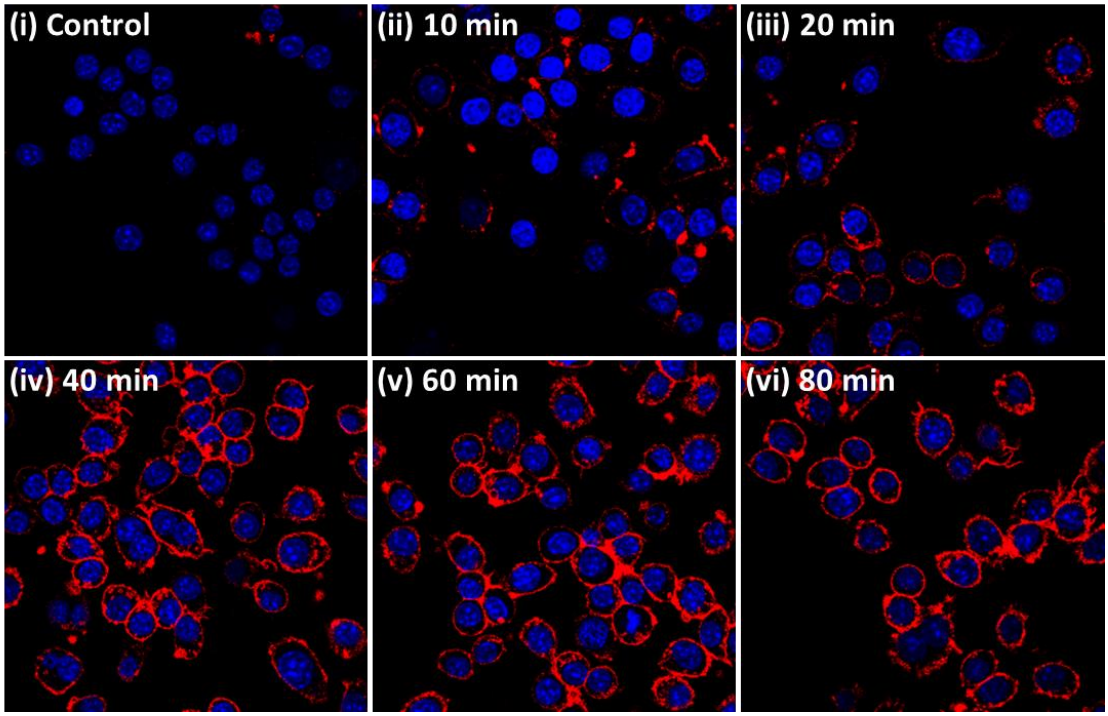


Fig. S12. Enlarged fluorescent images of **ONOO-CBP** labeled RAW264.7 cells in the (i) absence or presence of 1 μg/mL PMA after (ii) 10, (iii) 20, (iv) 40, (v) 60 and (vi) 80 minutes of incubation.

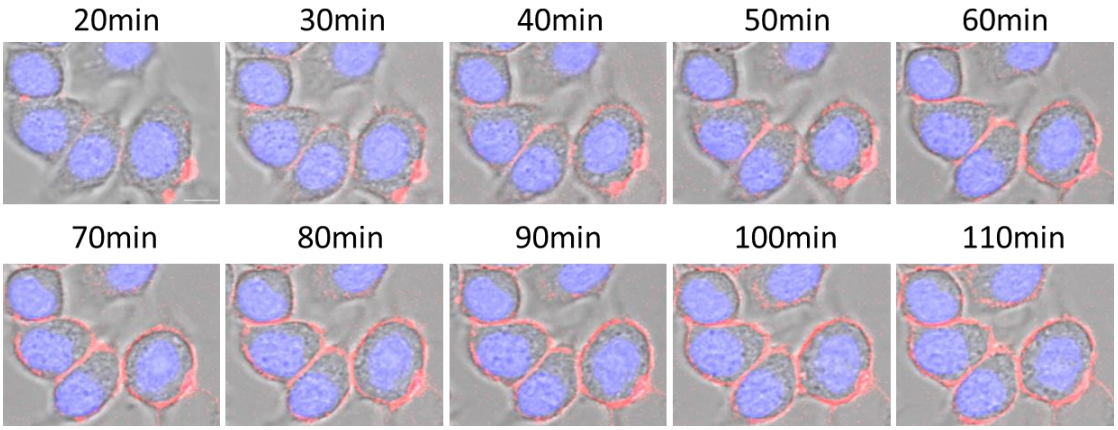
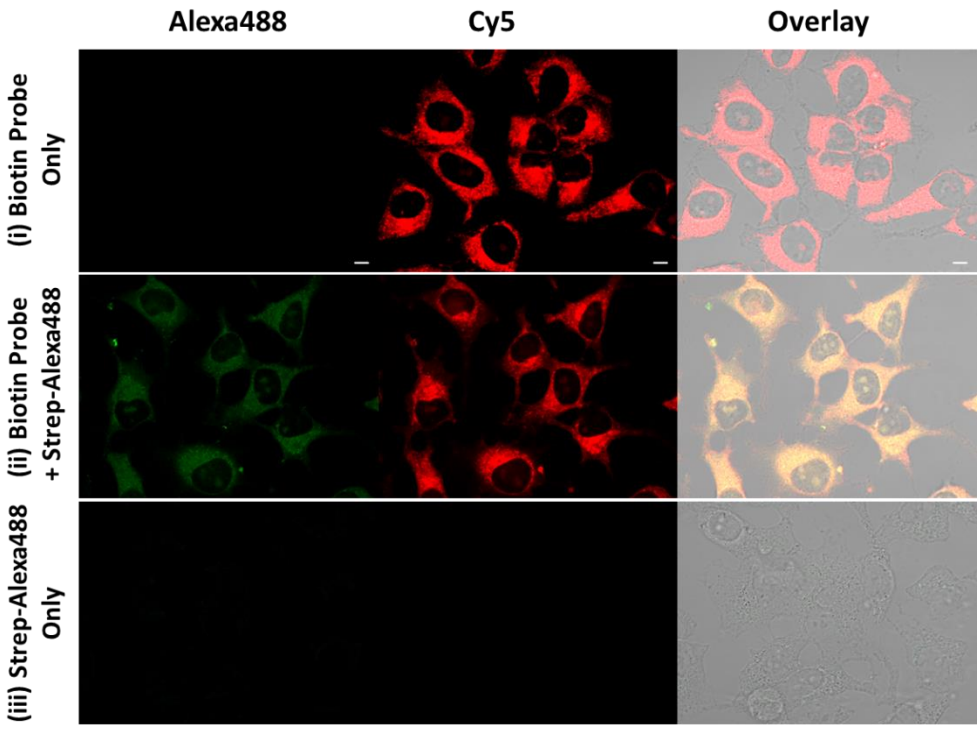


Fig. S13. Real-time tracking of PMA-stimulated macrophages without washing out the excess streptavidin-Cy5. The images show the overlay of Cy5, Hoechst 34580 and bright field images.

(a)



(b)

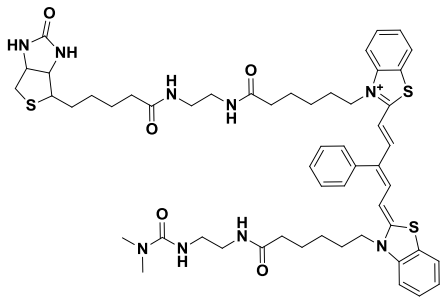
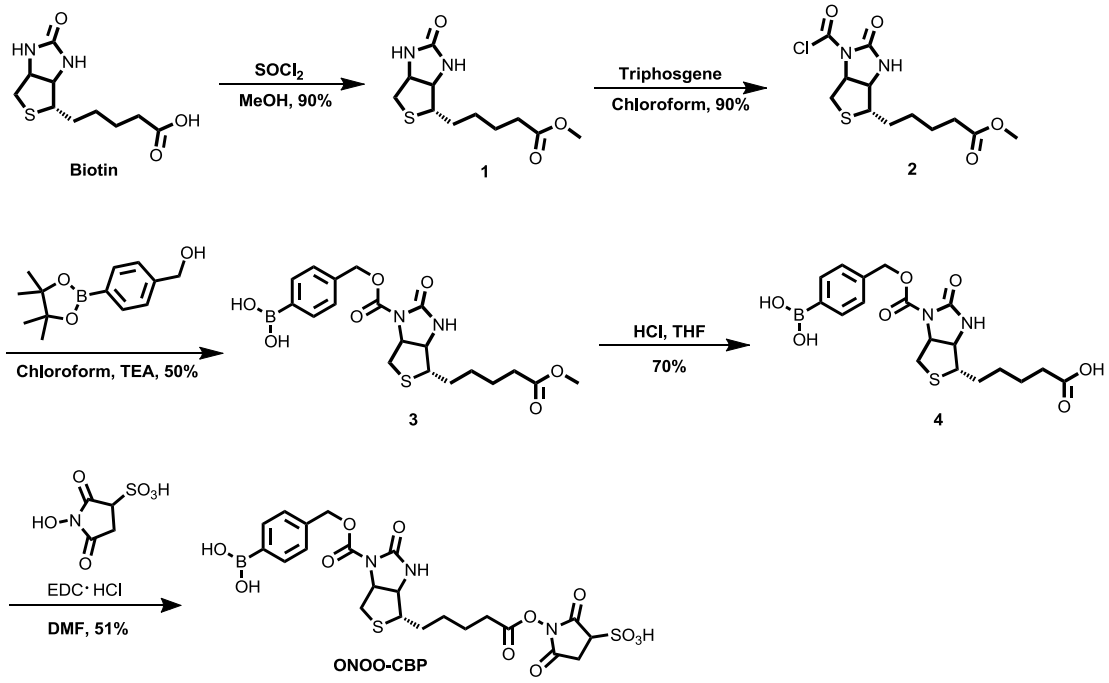
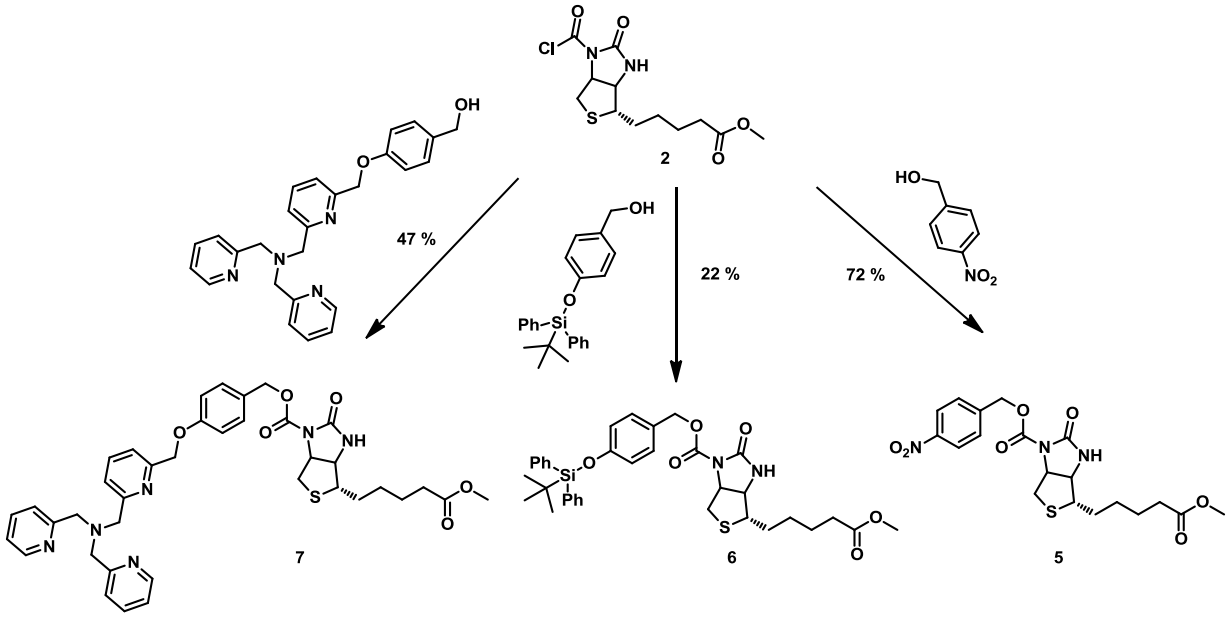


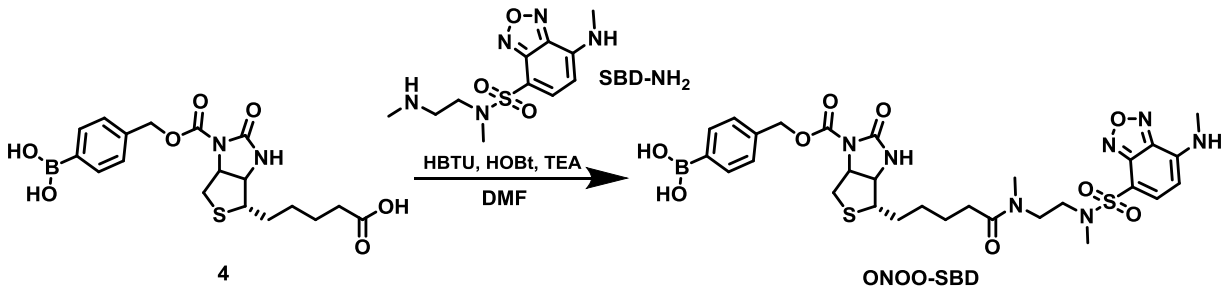
Fig. S14. Demonstration of potential application of streptavidin-biotin controlled binding probe for the detection of intracellular analytes. (a) Hela cells were incubated with 50 μM biotin probe for 30 minutes. After removing the excess probe, the cells were fixed with 4% paraformaldehyde. Streptavidin-Alexa488 was used to label the biotin probe. (i) biotin probe only, (ii) biotin probe and streptavidin-Alexa488 and (iii) streptavidin-Alexa488 only. (b) Chemical structure of the biotin probe **CK09** used in this study.



Scheme S1. Synthesis of cell-impermeable membrane-anchored CBP-biotin probe, **ONOO-CBP**, for the detection of secreted $ONOO^-$ on extracellular surface.



Scheme S2. Synthesis of CBP-biotin derivatives containing nitro, *tert*-butyldiphenylsilyl (TBDPS) and *tris*-[(2-pyridyl)-methyl]amine (TPA) functional groups.



Scheme S3. Synthesis of fluorogenic avidin-biotin controlled binding probe **ONOO-SBD**.

Synthesis of compound 1

To Biotin (0.5 g, 2.04 mmol) in 10 mL of dry MeOH was added dropwise thionyl chloride (0.97 mL, 6.5 mmol) in an ice bath. The solution was stirred for 12 hours at room temperature. After removing the solvent, the residue was redissolved in dichloromethane. The organic layer was extracted with $NaHCO_3$, brine and dried over $MgSO_4$, filtered and concentrated to give the compound **1** as a white solid. Yield = 90 %. R_f = 0.6 (DCM : MeOH =

9 : 1). **^{1}H NMR** (400 MHz, CD_3OD): δ 4.51-4.48 (m, 1H), 4.32-4.29 (m, 1H), 3.65 (s, 3H), 3.23-3.18 (m, 1H), 2.95-2.90 (m, 1H), 2.71 (d, *J* = 12.4 Hz, 1H), 2.35 (t, *J* = 7.4 Hz, 2H), 1.76-1.56 (m, 4H), 1.48-1.42 (m, 2H) ppm. **^{13}C NMR** (100 MHz, CD_3OD): δ 176.06, 166.28, 63.58, 61.83, 57.15, 52.22, 41.22, 34.74, 29.90, 29.65, 26.10 ppm. **ESI-MS**: m/z calc. for $C_{11}H_{19}N_2O_3S^+$ $[M+H]^+$, 259.1116; Found, 259.1111 $[M+H]^+$.

Synthesis of compound 2

To compound **1** (0.3 g, 1.16 mmol) in a reaction flask was added triphosgene (1.03g, 5.81mmol) in 18 mL dry chloroform at room temperature. The reaction mixture was refluxed at 80 °C for 24 hours. After removing the solvent, the residue was redissolved in ethyl acetate. The organic layer was extracted with water, brine and dried over $MgSO_4$, filtered and concentrated. The residue was purified by flash column chromatography (eluent: EA/HEX = 1/1) to afford compound **2** as a pale yellow oil. Yield = 90 %. R_f = 0.8 (EA/HEX = 5/1). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.16 (s, 1H), 4.94-4.91 (m, 1H), 4.24-4.21 (m, 1H), 3.63 (s, 3H), 3.20-3.13 (m, 2H), 3.06-3.01 (m, 1H), 2.31 (t, *J* = 7.4 Hz, 2H), 1.67-1.62 (m, 4H), 1.45-1.43 (m, 2H) ppm. **^{13}C NMR** (100 MHz, $CDCl_3$): δ 193.93, 154.10, 144.82, 64.60, 57.20, 55.00, 51.49, 38.29, 33.40, 28.08, 27.78, 24.47 ppm.

Synthesis of compound 3

To 4-(Hydroxymethyl)phenylboronic acid pinacol ester (393 mg, 1.68 mmol) in a reaction flask was added **2** (393 mg, 1.68 mmol) in 5 mL dry chloroform and triethylamine (468 μL, 3.36 mmol) at room temperature. The reaction mixture was refluxed at 80 °C for 18 hours. After removing the solvent, the residue was redissolved in ethyl acetate. The organic layer was extracted with water, brine and dried over $MgSO_4$, filtered and concentrated. The residue was purified by reverse-phase column chromatography (eluent: MeOH/H_2O = 3/1) to

afford compound **3** as a white solid. Yield = 50 %. R_f = 0.4 (EA : HEX = 5 : 1). **^{1}H NMR** (400 MHz, d-DMSO): δ 8.06 (s, 2H), 7.76 (d, *J* = 7.6 Hz, 2H), 7.71 (s, 1H), 7.34 (d, *J* = 7.6 Hz, 2H), 5.17-5.16 (m, 2H), 4.76-4.73 (m, 1H), 4.13-4.10 (m, 1H), 3.56 (s, 3H), 3.17-3.15 (m, 1H), 2.95-2.88 (m, 2H), 2.28 (t, *J* = 7.4 Hz, 2H), 1.58-1.45 (m, 4H), 1.33-1.30 (m, 2H) ppm. **^{13}C NMR** (100 MHz, d-DMSO): δ 173.98, 155.34, 151.78, 138.46, 134.84, 127.19, 67.35, 62.60, 57.90, 55.46, 51.89, 33.72, 28.65, 28.43, 25.05 ppm. **ESI-MS**: m/z calc. for $C_{19}H_{26}BN_2O_7S$ $[M+H]^+$, 437.1554; Found, 437.1568 $[M+H]^+$.

Synthesis of compound 4

To compound **3** (70mg, 0.16mmol) in 2 mL tetrahydrofuran was added dropwise 1 mL 3N HCl in ice bath. The solution was stirred for 24 hours at room temperature. After removing the solvent, the residue was purified by reversed-phase column chromatography (eluent: methanol/water = 1/1) to afford compound **4** as a white solid. Yield = 70%. R_f = 0.05 (EA : HEX = 5 : 1). **^{1}H NMR** (400 MHz, d-DMSO): δ 8.09 (br, 2H), 7.76 (d, *J* = 7.6 Hz, 2H), 7.71 (s, 1H), 7.34 (d, *J* = 7.6 Hz, 2H), 5.20-5.13 (m, 2H), 4.76-4.73 (m, 1H), 4.13-4.10 (m, 1H), 3.17-3.15 (m, 1H), 2.95-2.88 (m, 2H), 2.18 (t, *J* = 7.4 Hz, 2H), 1.62-1.42 (m, 4H), 1.32-1.30 (m, 2H) ppm. **^{13}C NMR** (125 MHz, d-DMSO): δ 174.38, 154.62, 151.10, 137.75, 134.14, 126.48, 66.65, 61.91, 57.23, 54.81, 33.42, 28.05, 27.80, 24.41 ppm. **ESI-MS**: m/z calc. for $C_{18}H_{22}BN_2O_7S^-$ $[M-H]^-$, 421.1241; Found, 421.1242 $[M-H]^-$.

Synthesis of ONOO-CBP

To compound **4** in 1.5 mL DMF was added N-Hydroxysulfosuccinimide sodium salt (9.2 mg, 0.043 mmol) and EDC·HCl (8.2 mg, 0.043 mmol). The solution was stirred for 16 hours at room temperature. The crude mixture was purified directly by reversed-phase preparative HPLC to give product **ONOO-CBP** as a white solid. Yield = 51 %. **^{1}H NMR** (400 MHz, D_2O):

7.77 (d, *J* = 7.6 Hz, 2H), 7.44 (d, *J* = 7.6 Hz, 2H), 5.25 (m, 2H), 4.46 (m, 1H), 4.28 (m, 1H), 3.31 (m, 2H), 3.19-3.14 (m, 1H), 3.01 (m, 2H), 2.69 (m, 2H), 1.73 (m, 3H), 1.47 (m, 3H) ppm. **^{13}C NMR** (125 MHz, D_2O): δ 173.10, 170.18, 168.92, 157.92, 137.97, 134.00, 127.37, 68.02, 62.87, 58.21, 56.48, 56.33, 54.63, 38.12, 30.15, 29.57, 27.45, 12.58 ppm.

Synthesis of compound 5

To 4-Nitrobenzyl alcohol (273 mg, 1.78 mmol) in dry chloroform (10 mL) was added Triethylamine (331 µL, 2.38 mmol) and compound **2** (200 mg, 0.59 mmol) under argon at room temperature. The reaction mixture was refluxed at 80 °C for 18 hours. After removing the solvent, the residue was redissolved in dichloromethane (30 mL) and extracted with water (30 mL). The combined organic layer was washed with brine (30 mL), dried over anhydrous sodium sulfate, filtered and concentrated. The crude residue was purified by flash column chromatography to give the desired product **5** as a white powder (187 mg, 72%); **^{1}H NMR** (500 MHz, DMSO-d_6): δ 8.23 (d, *J* = 8.5Hz, 2H), 7.77 (s, 1H), 7.69 (d, *J* = 8.5 Hz, 2H), 5.33 (s, 2H), 4.79-4.77 (m, 1H), 4.16-4.13 (m, 1H), 3.57 (s, 3H), 3.20-3.18 (m,1H), 3.01-2.93 (m, 2H), 2.29 (t, *J* = 7.5Hz, 2H), 1.63-1.28 (m, 6H) ppm; **^{13}C NMR** (125 MHz, DMSO-d_6): δ 173.29, 154.58, 150.94, 147.02, 143.94, 128.12, 123.55, 65.55, 61.92, 57.32, 54.75, 51.23, 51.19, 37.99, 33.05, 27.98, 27.76, 24.38 ppm; **ESI-MS**: m/z calc. for $C_{19}H_{24}N_3O_7S^+$ $[M+H]^+$, 438.1335; Found, 438.1331 $[M+H]^+$.

Synthesis of compound 6

To a stirred solution of TBDPS-benzyl alcohol (0.5 g, 1.4 mmol) in dry chloroform (30 mL) was added compound **2** (0.97 g, 3 mmol) and triethylamine (0.96 mL, 6.9 mmol) under argon at room temperature. The reaction mixture was refluxed at 80 °C for 18 hours. After removing the solvent, the residue was redissolved in dichloromethane (30 mL) and extracted with water (30 mL). The combined organic layer was washed with brine (30 mL), dried over

anhydrous sodium sulfate, filtered and concentrated. The crudes were purified by column chromatography (eluent: EA : Hex = 1 : 3) to give the desired product **6** as a colorless oil. Yield = 22 % (199 mg); **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.73 – 7.65 (m, 4H), 7.44 – 7.31 (m, 6H), 7.12 (d, J = 8.4 Hz, 2H), 6.71 (d, J = 8.4 Hz, 2H), 6.59 (s, 1H), 5.22 – 4.99 (m, 2H), 4.80 – 4.66 (m, 1H), 4.26 – 4.07 (m, 1H), 3.61 (s, 3H), 3.16 – 3.07 (m, 1H), 3.05 – 2.83 (m, 2H), 2.27 (t, J = 7.2 Hz, 2H), 1.73 – 1.52 (m, 4H), 1.46 – 1.30 (m, 2H), 1.07 (s, 9H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$): δ 173.82, 155.97, 155.56, 151.22, 135.31, 132.52, 129.93, 129.80, 129.50, 127.83, 127.65, 119.67, 119.54, 77.32, 77.00, 76.68, 67.51, 62.47, 57.69, 55.04, 51.34, 38.54, 33.39, 28.10, 27.81, 26.33, 24.39, 19.27 ppm; **ESI-MS**: m/z calc. for $C_{35}H_{42}N_2NaO_6SSi$ $[M+Na]^+$ 669.2430, found 669.2436 $[M+Na]^+$.

Synthesis of compound 7

To a stirred solution of TPA-benzyl alcohol (0.2 g, 0.47 mmol) in dry chloroform (15 mL) was added compound **16** (0.6 g, 1.86 mmol) and triethylamine (131 μL, 0.94 mmol) under argon at room temperature. The reaction mixture was refluxed at 80 $^{\circ}$C for 18 hours. After removing the solvent, the residue was redissolved in dichloromethane (30 mL) and extracted with water (30 mL). The combined organic layer was washed with brine (30 mL), dried over anhydrous sodium sulfate, filtered and concentrated. The crude product was purified by column chromatography (eluent: DCM : MeOH = 9 : 1) to give the desired product **7** as a dark green oil. **Yield** = 47 % (157 mg); **^{1}H NMR** (400 MHz, $CDCl_3$): δ 8.50 (d, J = 4.4 Hz, 1H), 7.66 – 7.60 (m, 3H), 7.57 – 7.42 (m, 3H), 7.35 – 7.28 (m, 3H), 7.15 – 7.06 (m, 1H), 6.92 (d, J = 8.0 Hz, 1H), 6.11 (s, 1H), 5.27 – 5.07 (m, 4H), 4.79 – 4.71 (m, 1H), 4.17 – 4.09 (m, 1H), 3.87 (s, 3H), 3.66 – 3.58 (m, 1H), 3.45 (d, J = 1.1 Hz, 1H), 3.19 – 3.03 (m, 2H), 3.03 – 2.88 (m, 1H), 2.29 (t, J = 7.6 Hz, 1H), 1.73 – 1.55 (m, 2H), 1.45 – 1.32 (m, 4H) ppm. (2 forms); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 173.54, 158.82, 158.56, 158.11, 155.89, 155.67, 151.06, 148.65, 136.90,

136.13, 129.69, 128.07, 127.65, 122.53, 121.69, 121.39, 119.14, 114.42, 114.29, 67.21, 63.73, 62.20, 59.73, 59.65, 57.47, 54.89, 51.09, 45.48, 38.31, 33.13, 27.89, 27.64, 24.16 ppm. (2 forms)

Synthesis of ONOO-SBD

To a stirred solution of Compound **4** (15 mg, 0.035 mmol), HBTU (19.90 mg, 0.525mmol), HOBt (6.96 mg, 0.046 mmol), TEA (240 mL, 0.175 mmol) in DMF (2 mL) was added **SBD-NH_2** (15.7 mg, 0.525 mmol) at room temperature. The resulting mixture was stirred overnight at room temperature. The reaction mixture was extracted with DCM (15 mL X 3) and the combined organic layers were dried over anhydrous sodium sulfate, filtered and concentrated. The crude product was purified by C18 reverse-phase column chromatography (H_2O/MeOH as eluent) to give the desired product **ONOO-SBD** as a yellow powder after lyophilization. **Yield** = 85 % (21 mg); **^{1}H NMR** (500 MHz, $CDCl_3$): δ 8.38-8.34 (m, 1H), 8.09-8.08 (m, 2H), 7.90-7.85 (m, 1H), 7.78 (d, *J* = 8.0 Hz, 2H), 7.71-7.70 (m, 1H), 7.36 (d, *J* = 8.0 Hz, 2H), 6.22-6.20 (m, 1H), 5.22-5.16 (m, 2H), 4.78-4.76 (m, 1H), 4.16-4.14 (m, 1H), 3.49-3.42 (m, 2H), 3.29-3.19 (m, 3H), 3.01-2.97 (m, 6H), 2.93-2.90 (m, 1H), 2.80-2.75 (m, 4H), 2.32-2.22 (m, 2H), 1.66-1.65 (m, 1H), 1.51-1.47 (m, 3H), 1.36-1.35 (m, 2H) ppm; **^{13}C NMR** (125 MHz, $CDCl_3$): δ 172.22, 171.98, 154.67, 151.14, 145.95, 144.33, 142.09, 141.93, 140.29, 140.00, 137.79, 134.17, 126.51, 107.00, 106.53, 98.34, 66.69, 61.96, 57.26, 54.93, 48.06, 47.79, 47.23, 44.74, 38.10, 35.46, 35.36, 34.61, 33.07, 32.33, 31.58, 29.76, 28.35, 28.22, 27.94, 24.86, 24.46 ppm (2 forms); **ESI-MS**: m/z calc. for $C_{29}H_{39}BN_7O_9S_2$ $[M+H]^+$ 704.2343, found 704.2326 $[M+H]^+$.

YP99
YP99

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8	Author	yp820805
9	Solvent	cd3od
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11	Pulse Sequence	s2pul
12	Experiment	1D
13	Number of Scans	12
14	Receiver Gain	30
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16	Pulse Width	0.0000
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18	Acquisition Date	2017-05-03T16:07:19
19	Modification Date	2017-05-03T16:10:00
20	Spectrometer Frequency	400.44
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23	Nucleus	1H
24	Acquired Size	11982
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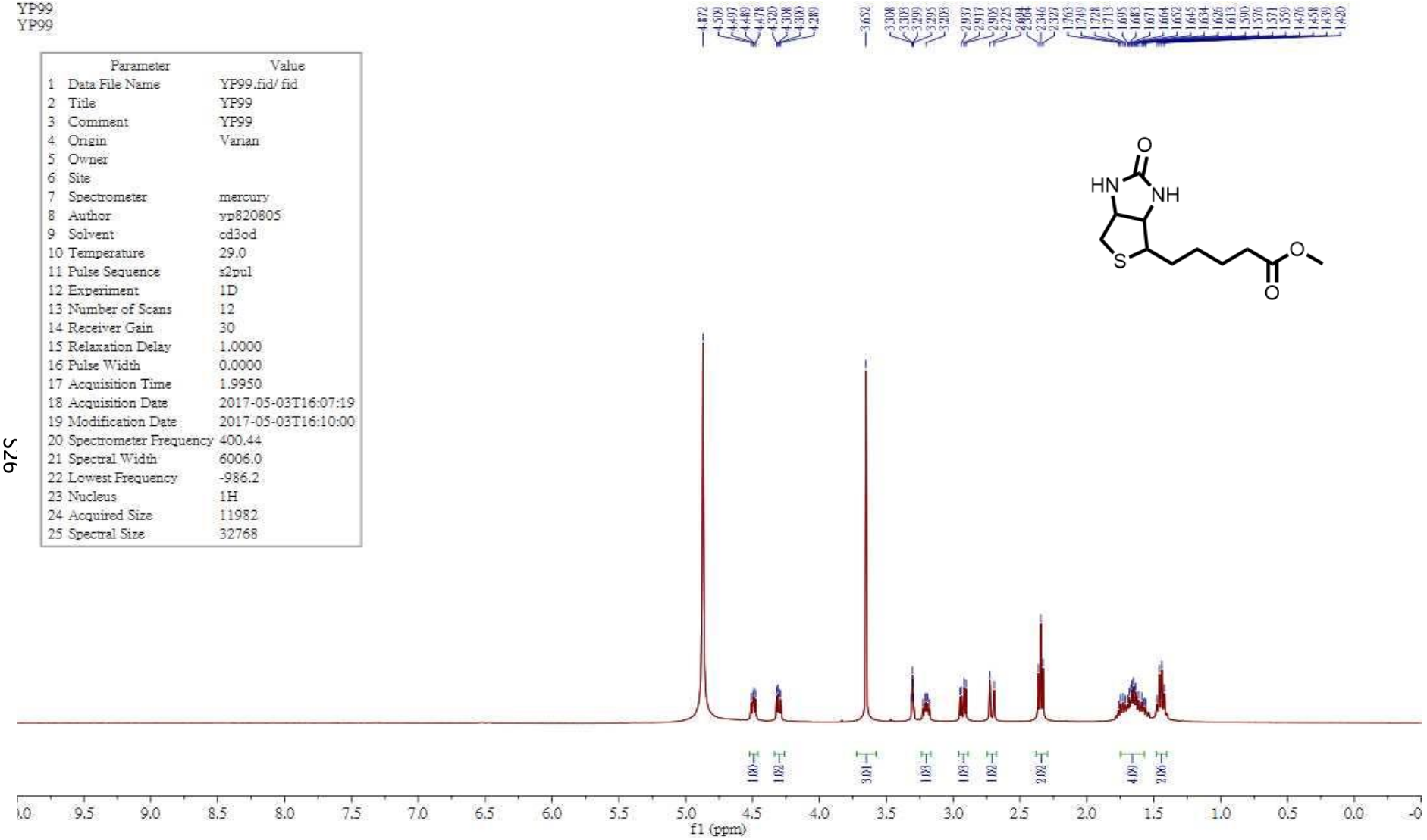

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YP99-C

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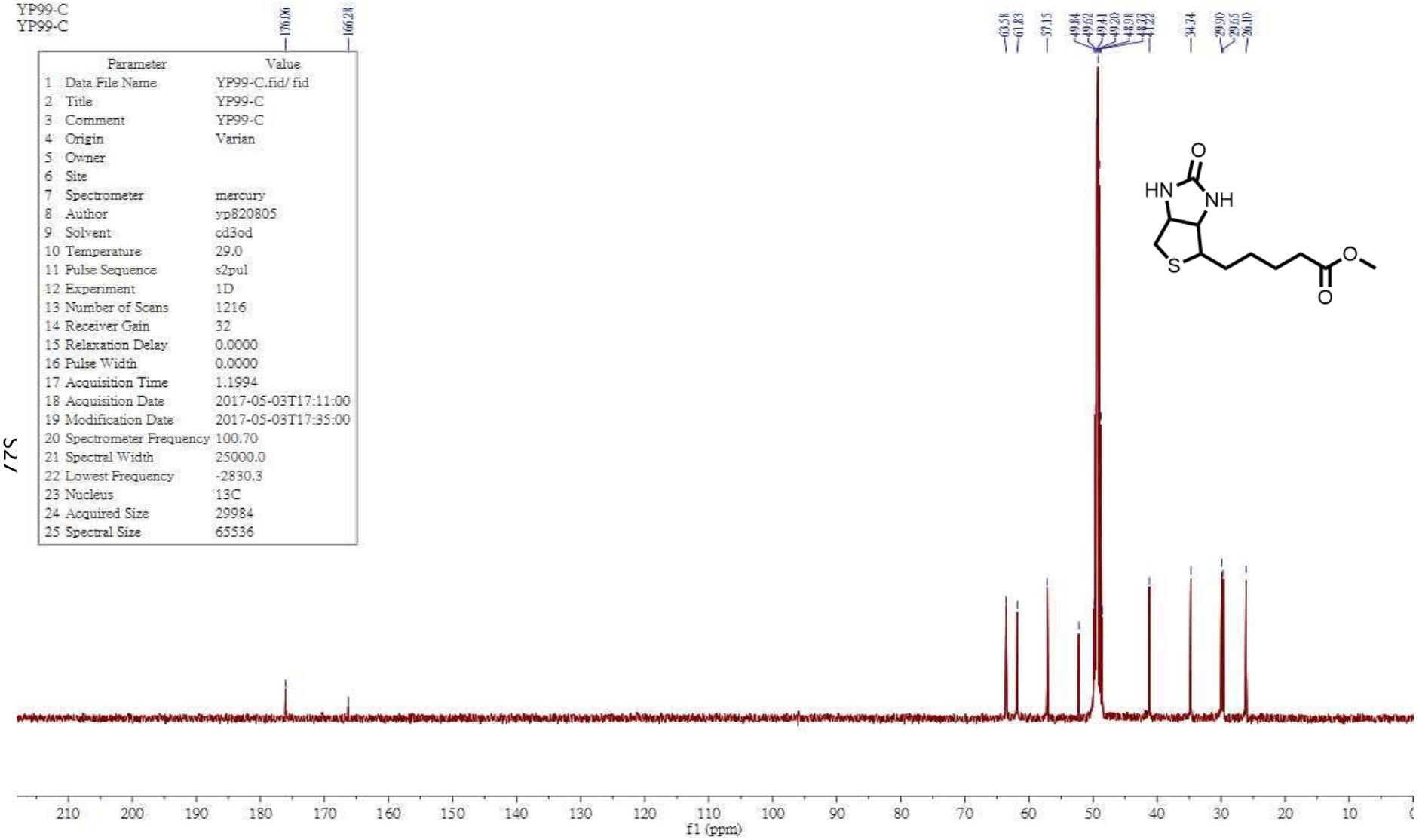
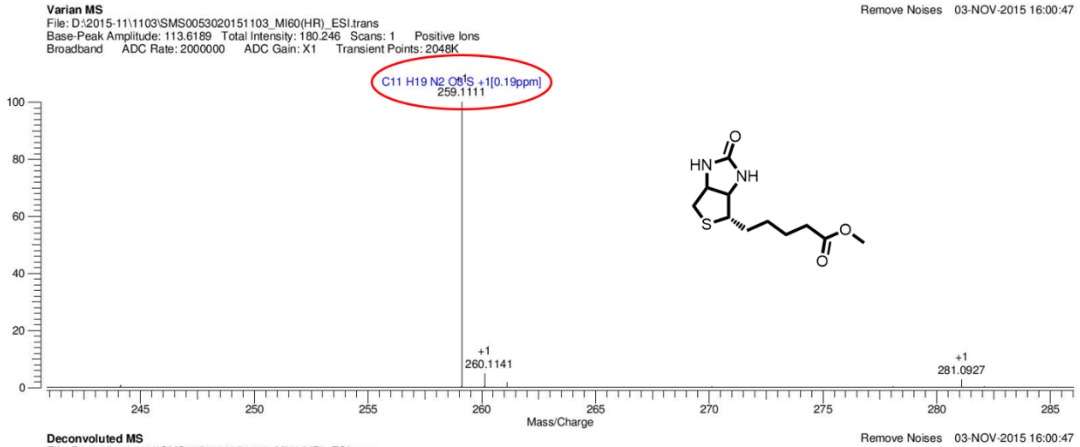
24	Acquired Size	29984
25	Spectral Size	65536

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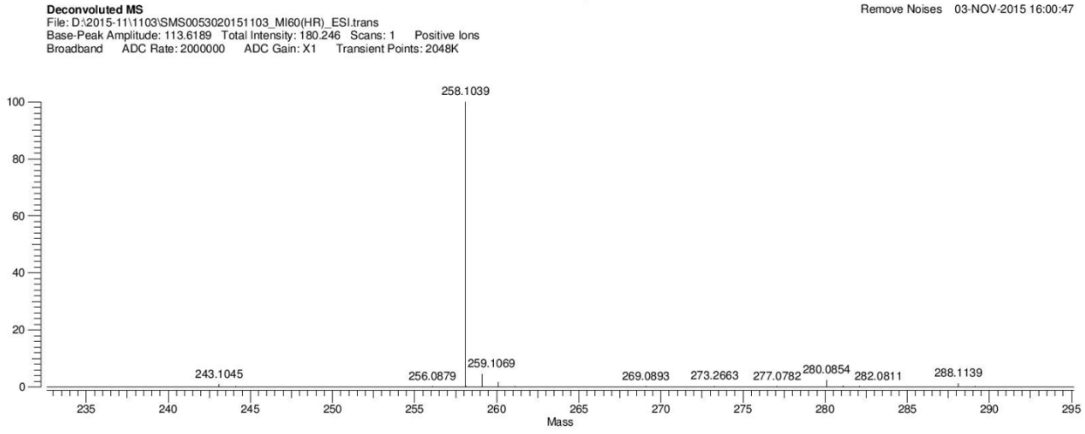
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Broadband ADC Rate: 2000000 ADC Gain: X1 Transient Points: 2048K
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YP60

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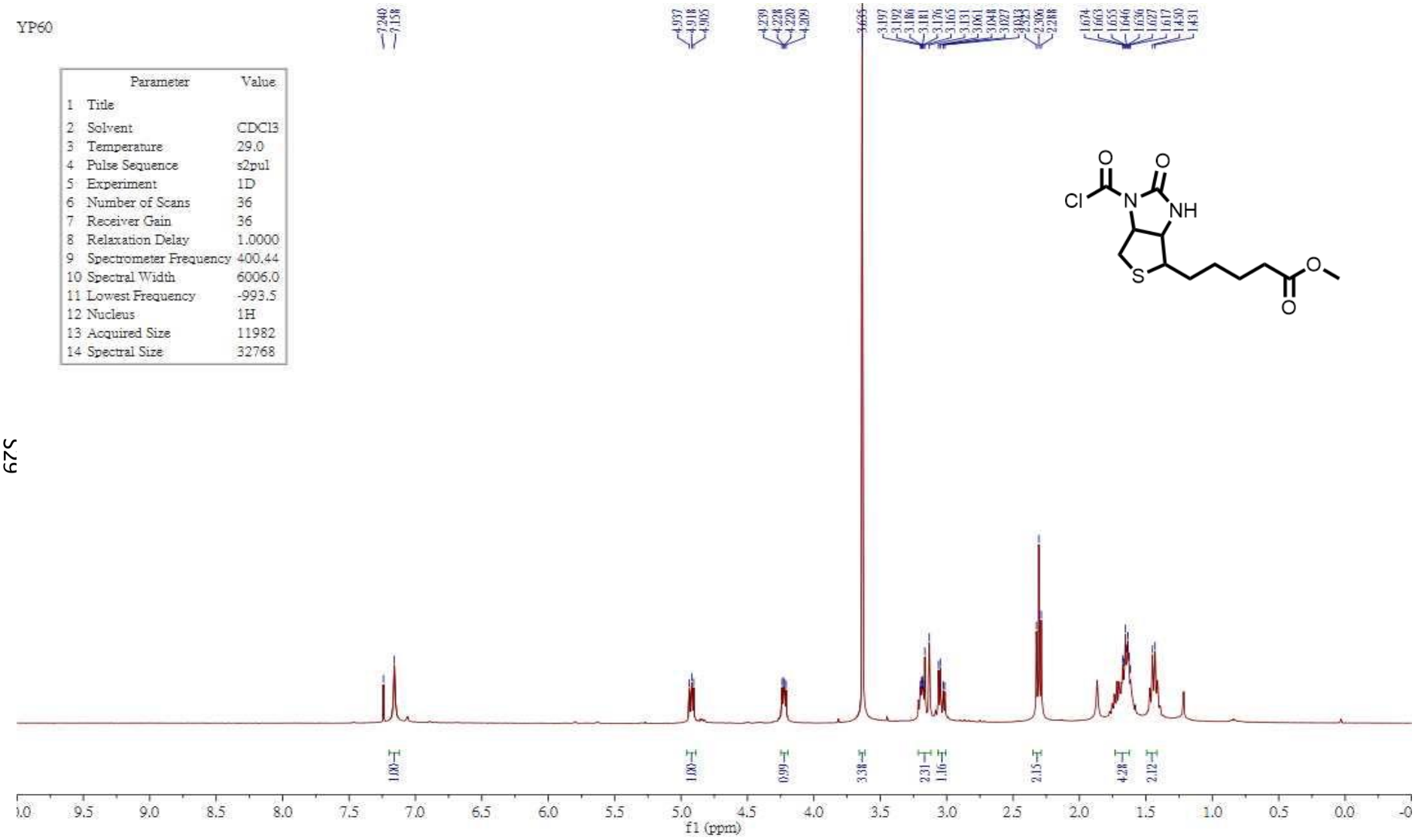

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YP60

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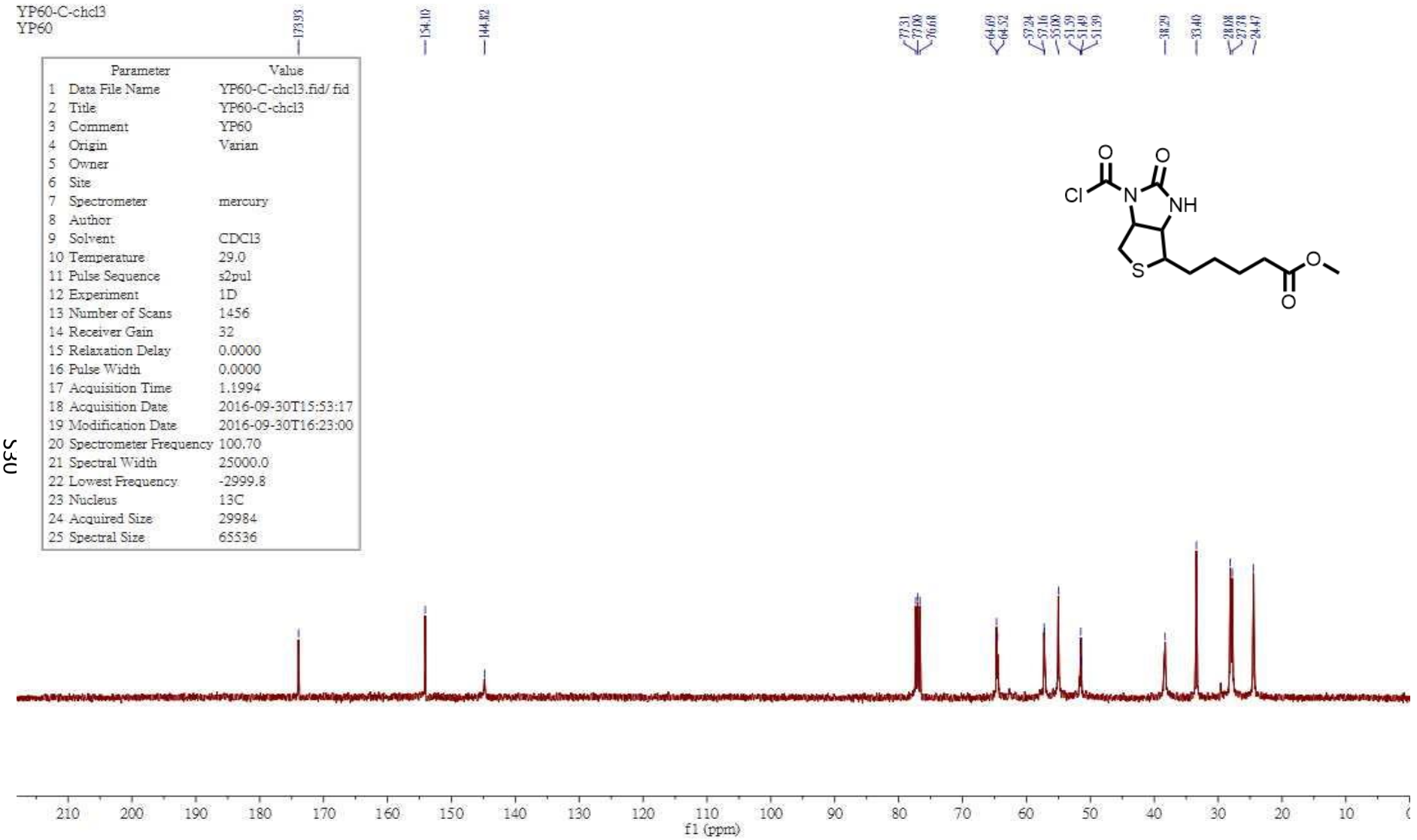

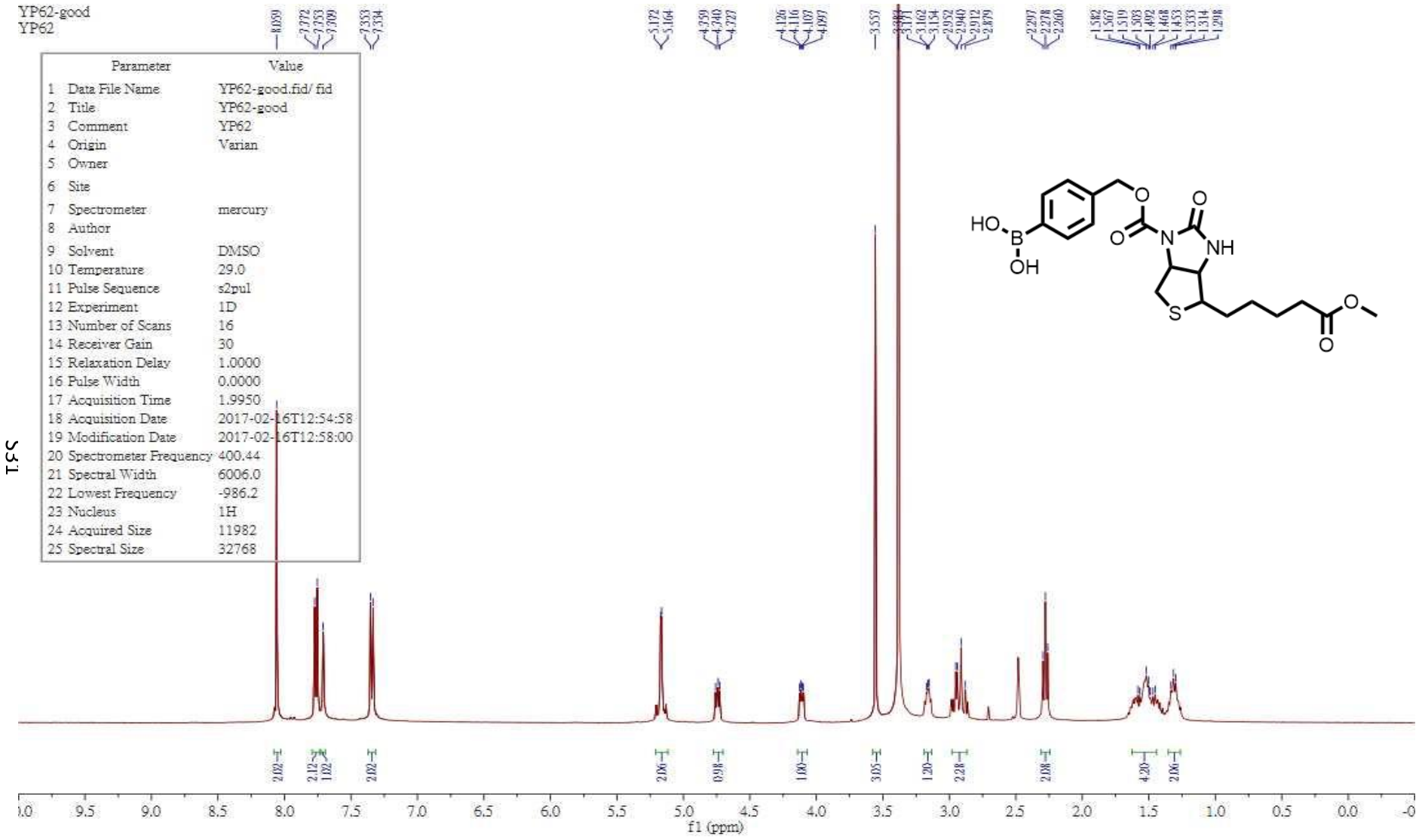
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YP62
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7 Spectrometer mercury
8 Author
9 Solvent DMSO
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12 Experiment 1D
13 Number of Scans 16
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NH
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S
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f1 (ppm)

YP62-C
YP62

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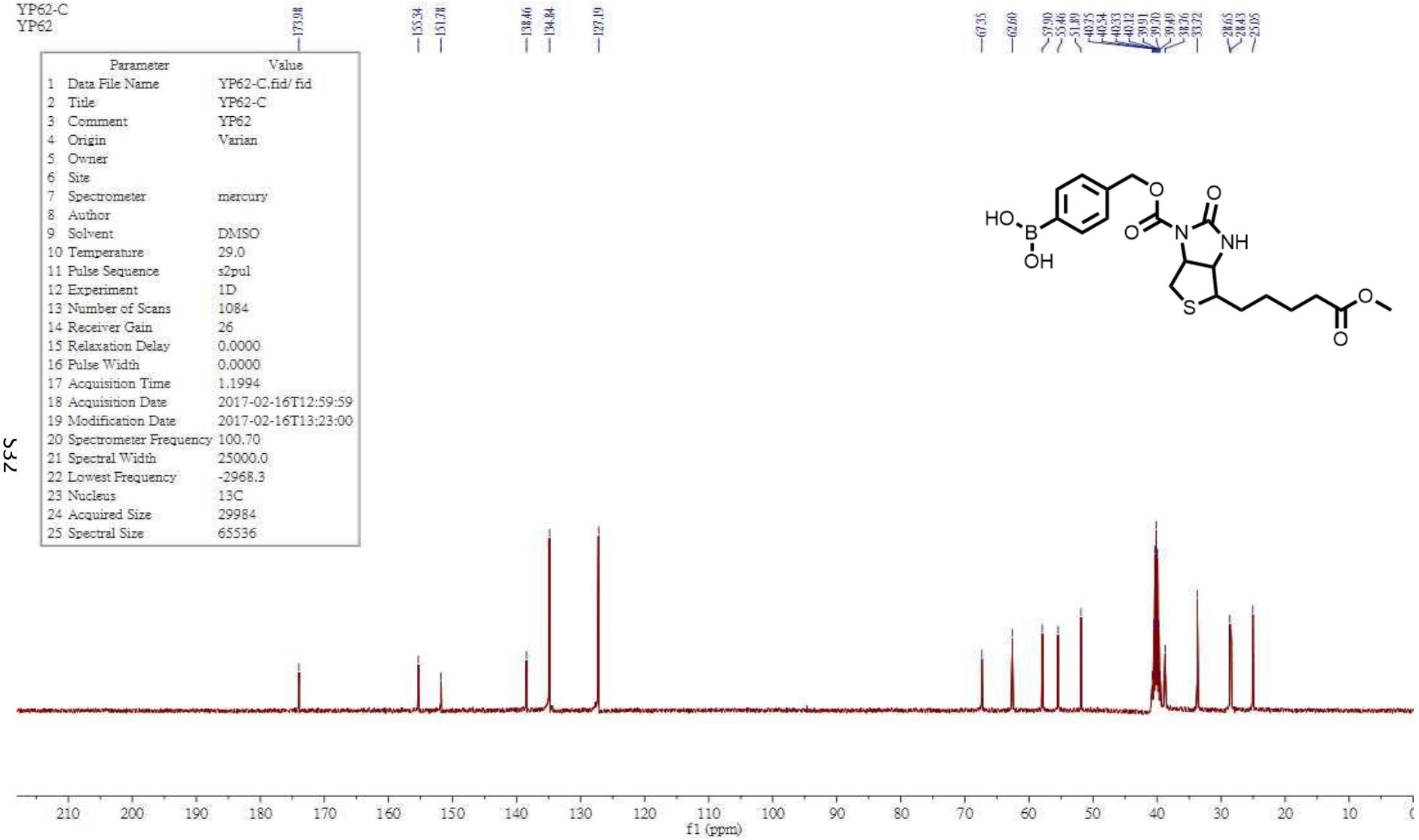

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Description:
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Created by:AccuTOF

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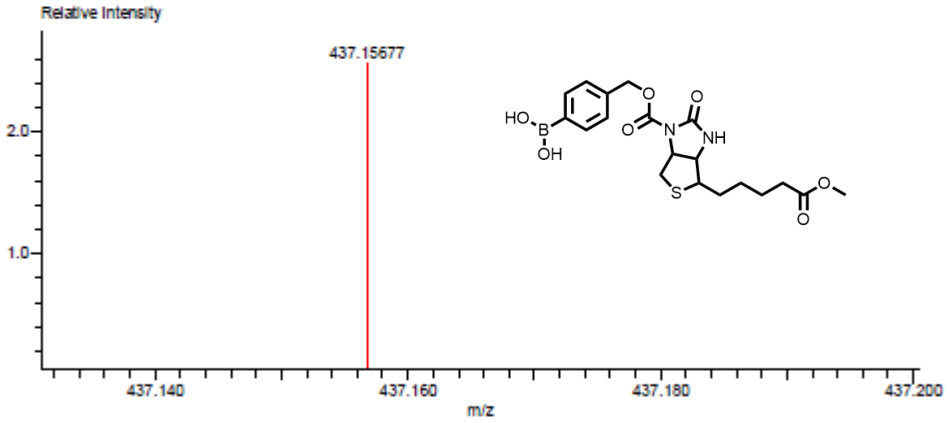

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YP71-1220-good
YP71

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8	Author	
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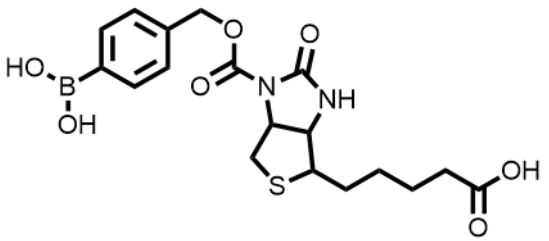

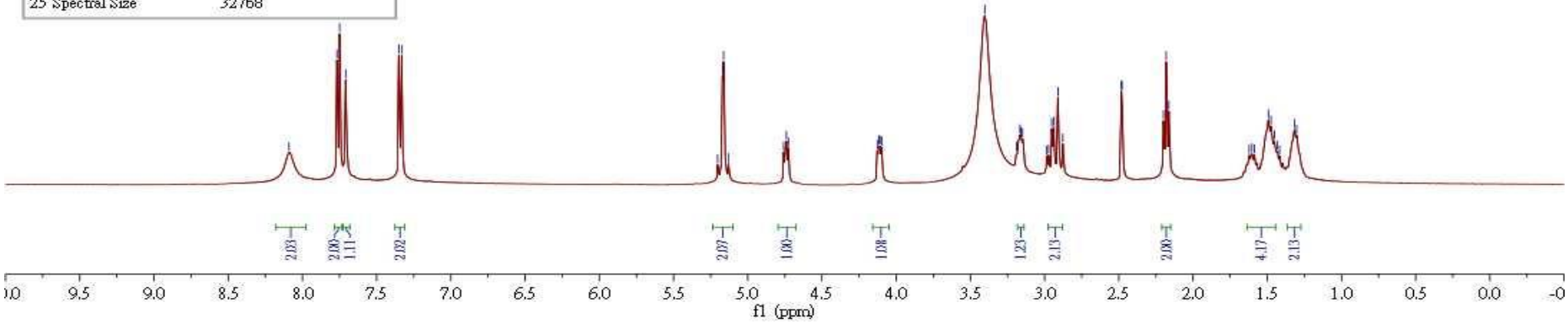

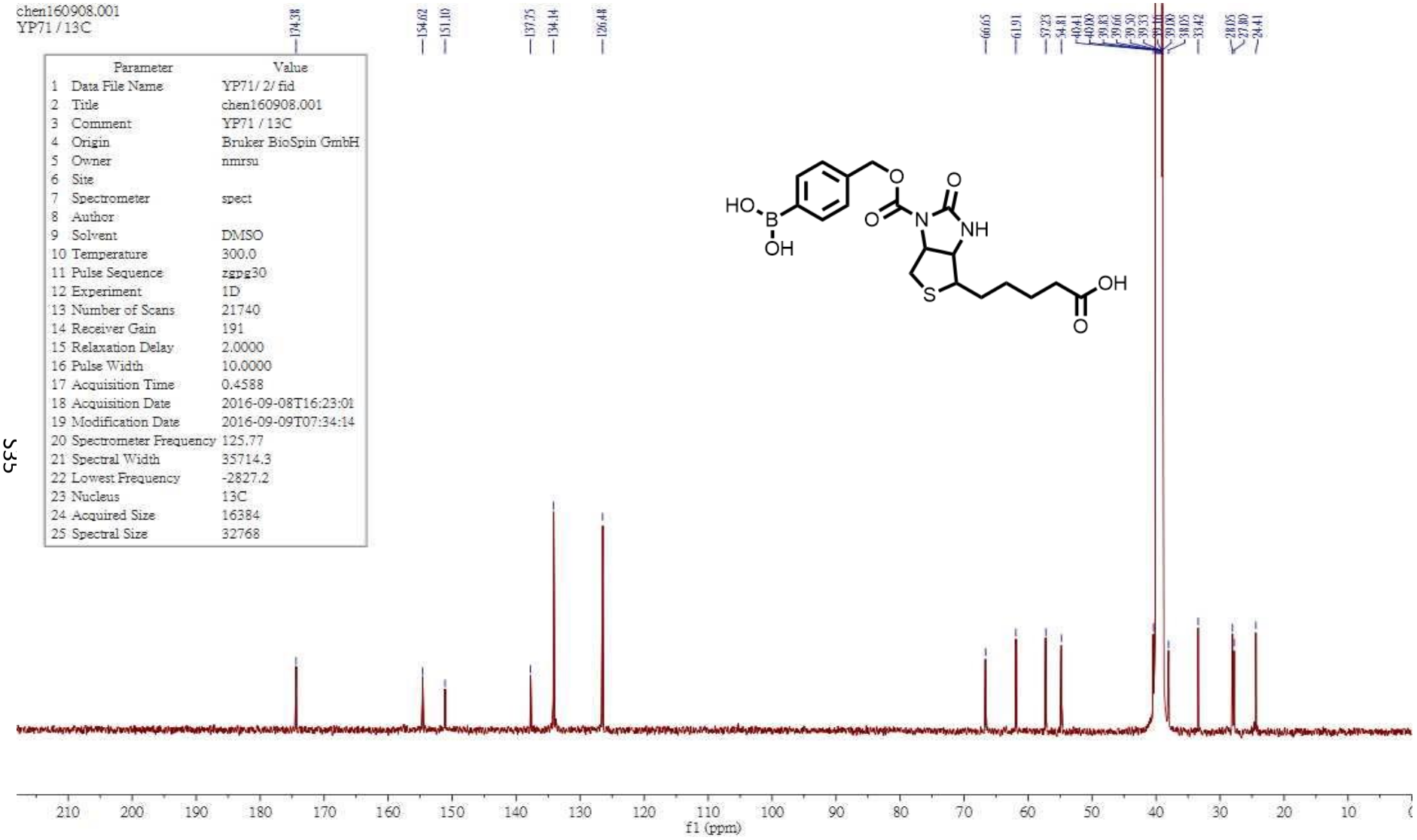

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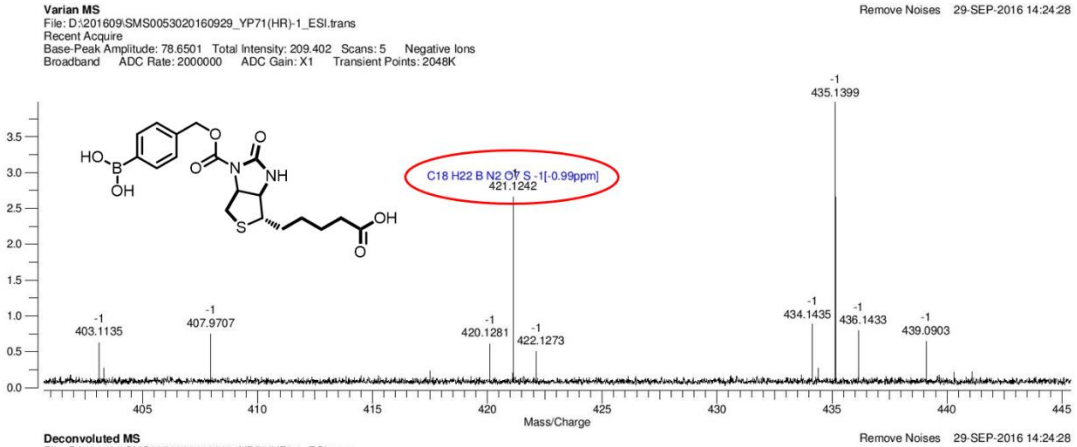
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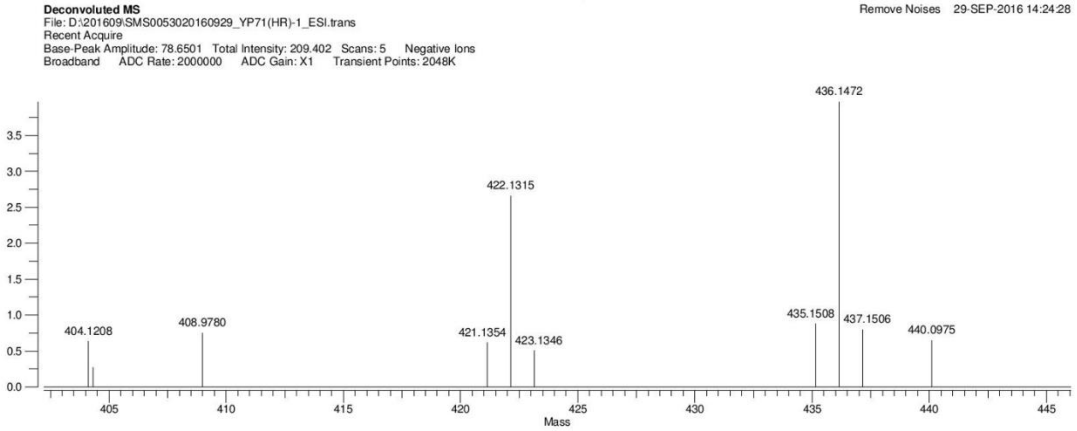
Mass/Charge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Broadband ADC Rate: 2000000 ADC Gain: X1 Transient Points: 2048K
Remove Noises 29-SEP-2016 14:24:28
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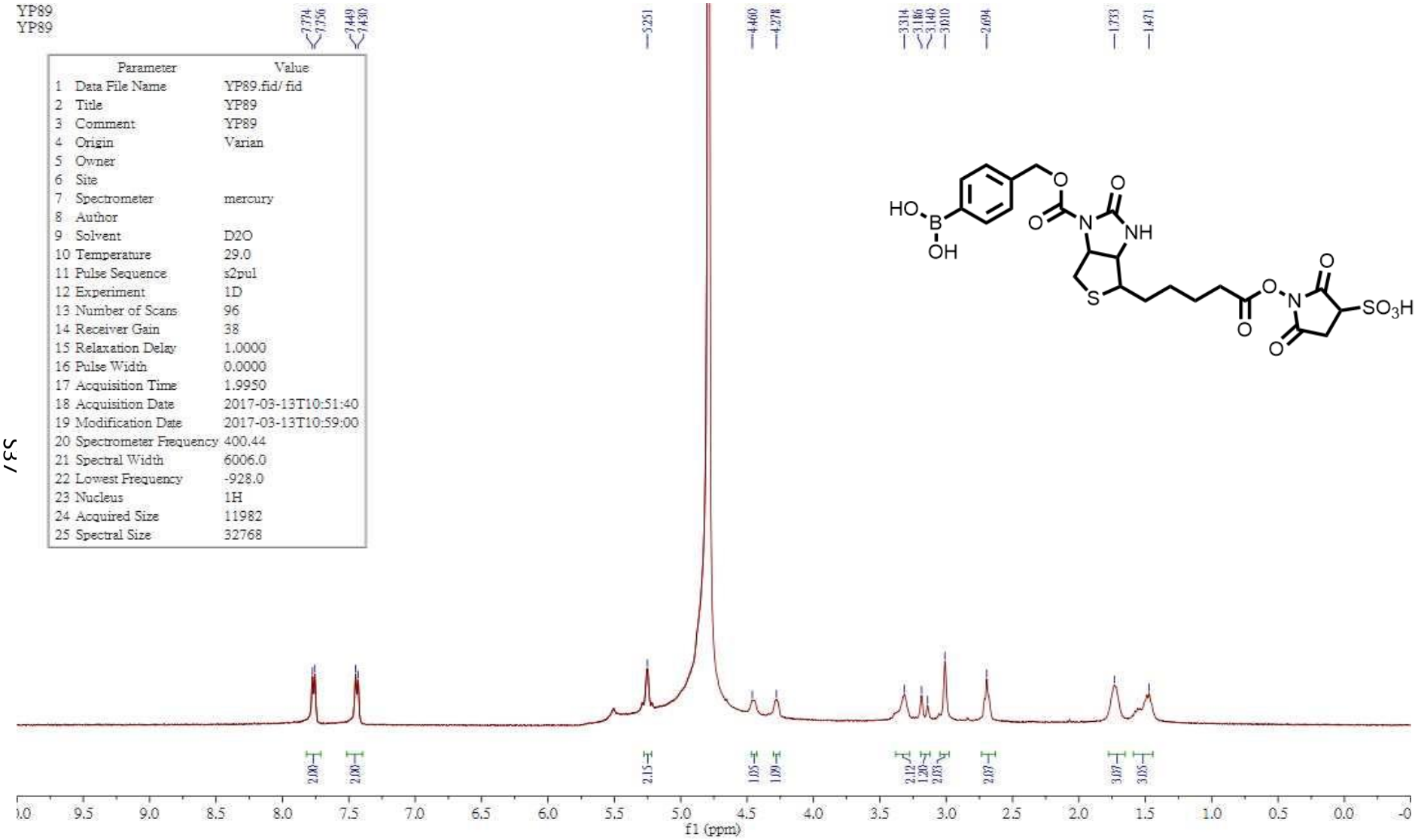
Mass



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25	Spectral Size	32768

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YP89

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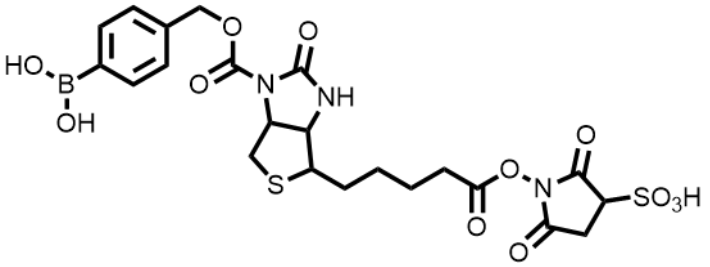
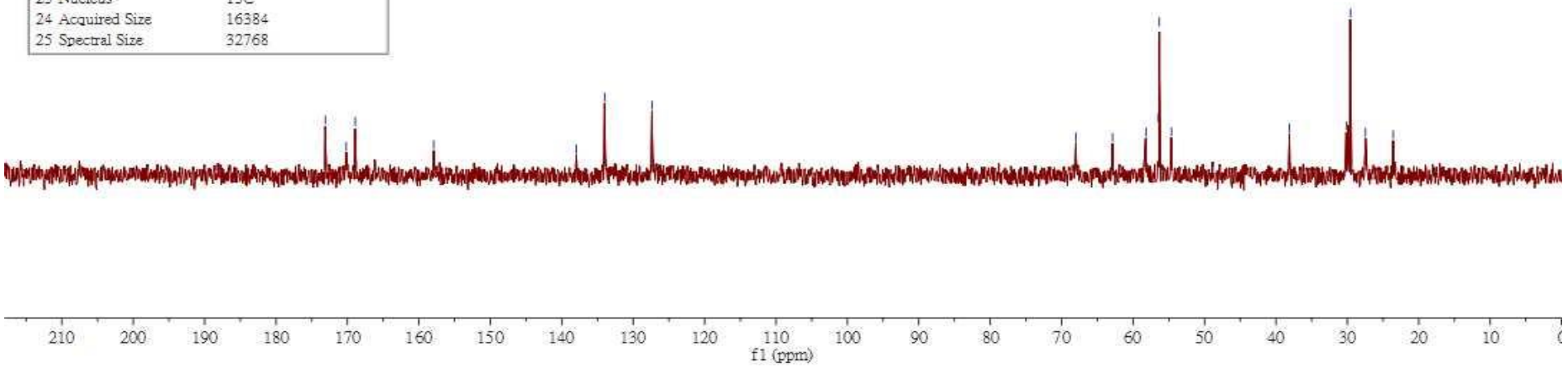
157.92
137.97
134.88
127.37
68.02
62.87
58.21
56.48
56.33
54.63
38.12
30.15
29.57
27.45
23.58

	Parameter	Value
1	Data File Name	YP89/2/fid
2	Title	chen170313.001
3	Comment	YP89
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	D2O
10	Temperature	299.6
11	Pulse Sequence	zgpg30
12	Experiment	1D
13	Number of Scans	20925
14	Receiver Gain	191
15	Relaxation Delay	2.0000
16	Pulse Width	10.0000
17	Acquisition Time	0.5505
18	Acquisition Date	2017-03-14T07:34:05
19	Modification Date	2017-03-14T09:15:04
20	Spectrometer Frequency	125.77
21	Spectral Width	29761.9
22	Lowest Frequency	-1675.6
23	Nucleus	13C
24	Acquired Size	16384
25	Spectral Size	32768

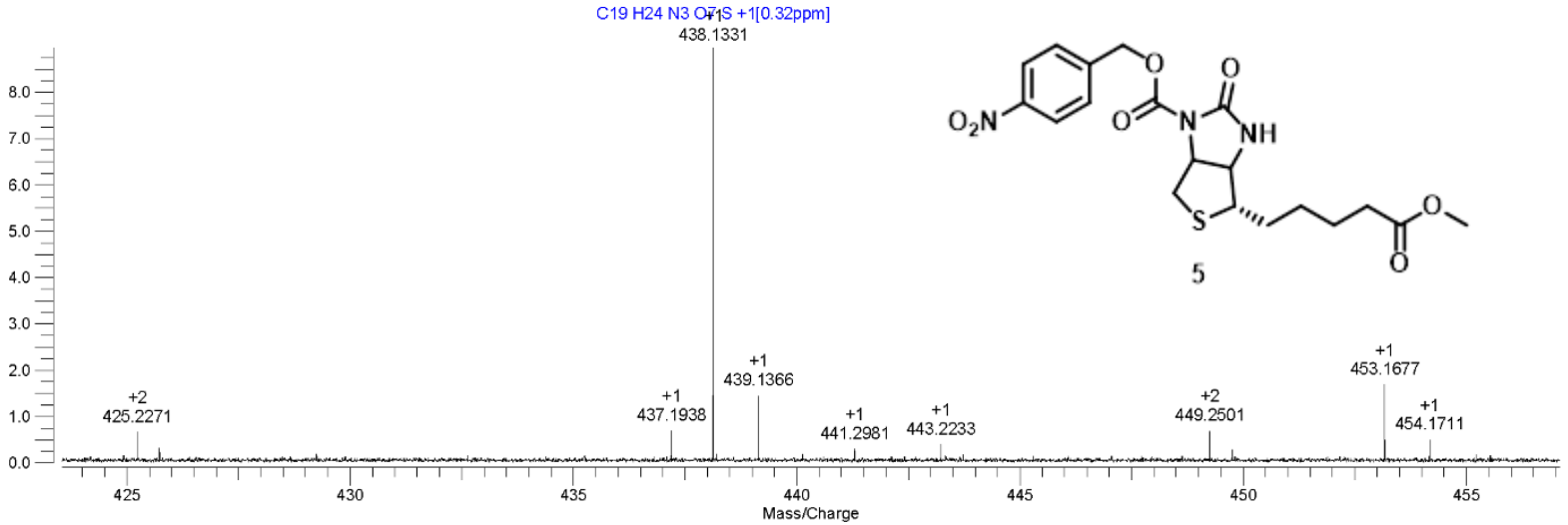




Varian MS

File: D:\2015-11\1103\SMS0053020151103_MI61(HR)_ESI.trans
Base-Peak Amplitude: 48.6073 Total Intensity: 391.211 Scans: 1 Positive Ions
Broadband ADC Rate: 2000000 ADC Gain: X1 Transient Points: 2048K

Remove Noises 03-NOV-2015 16:08:11

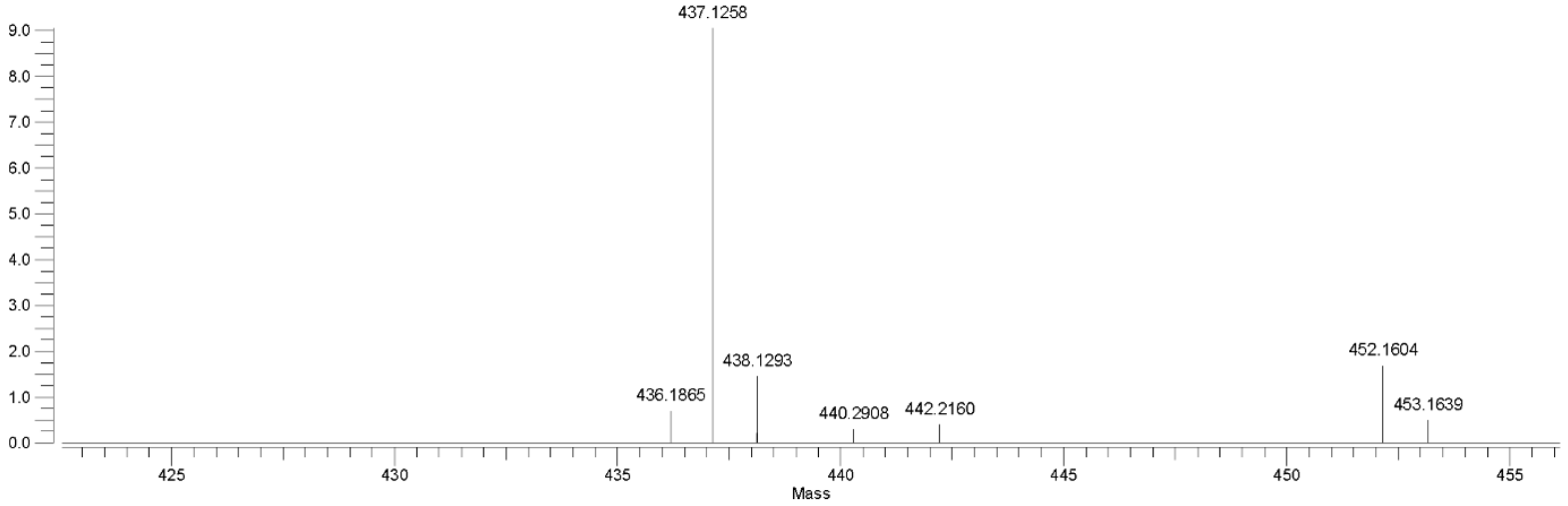


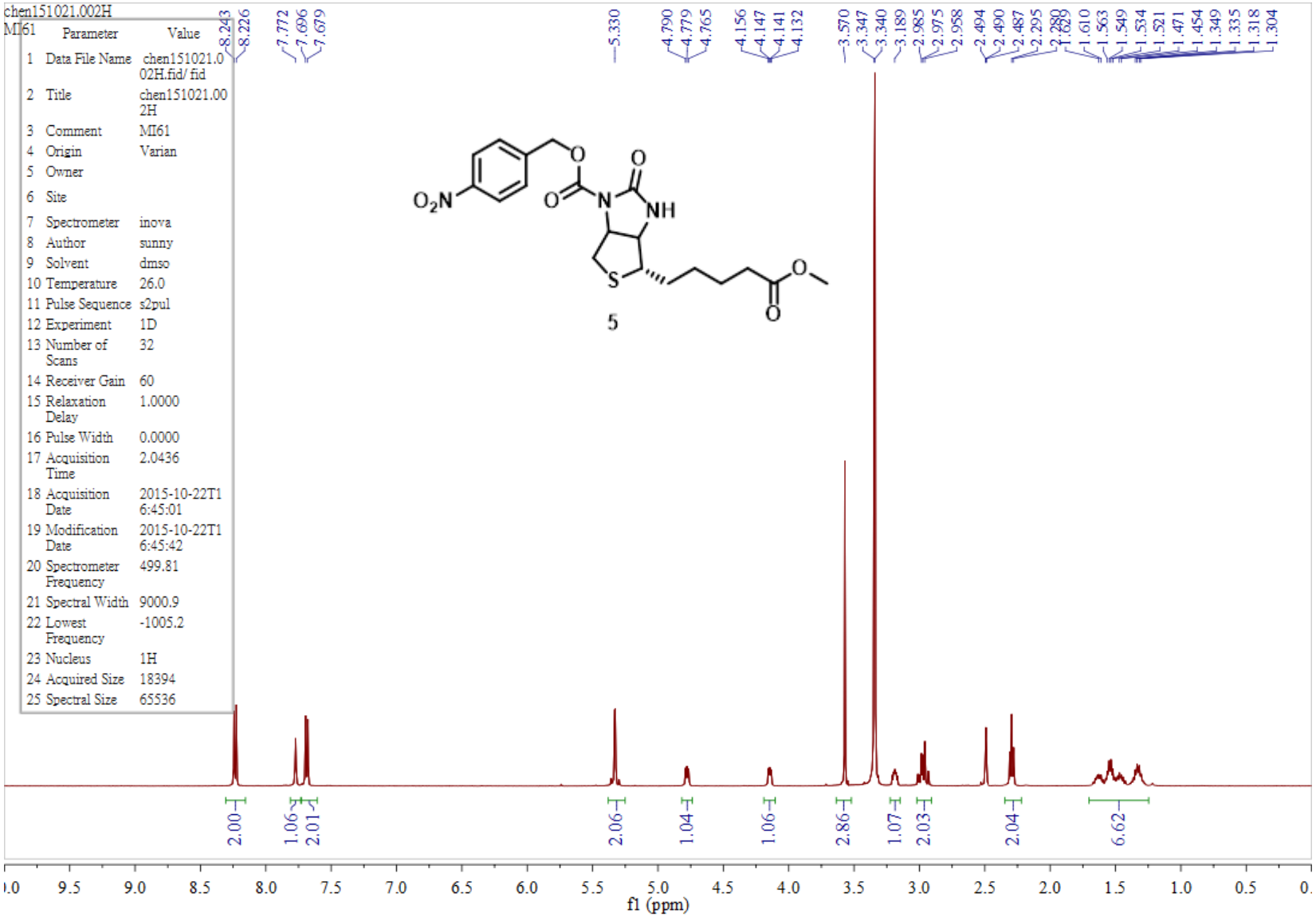
5

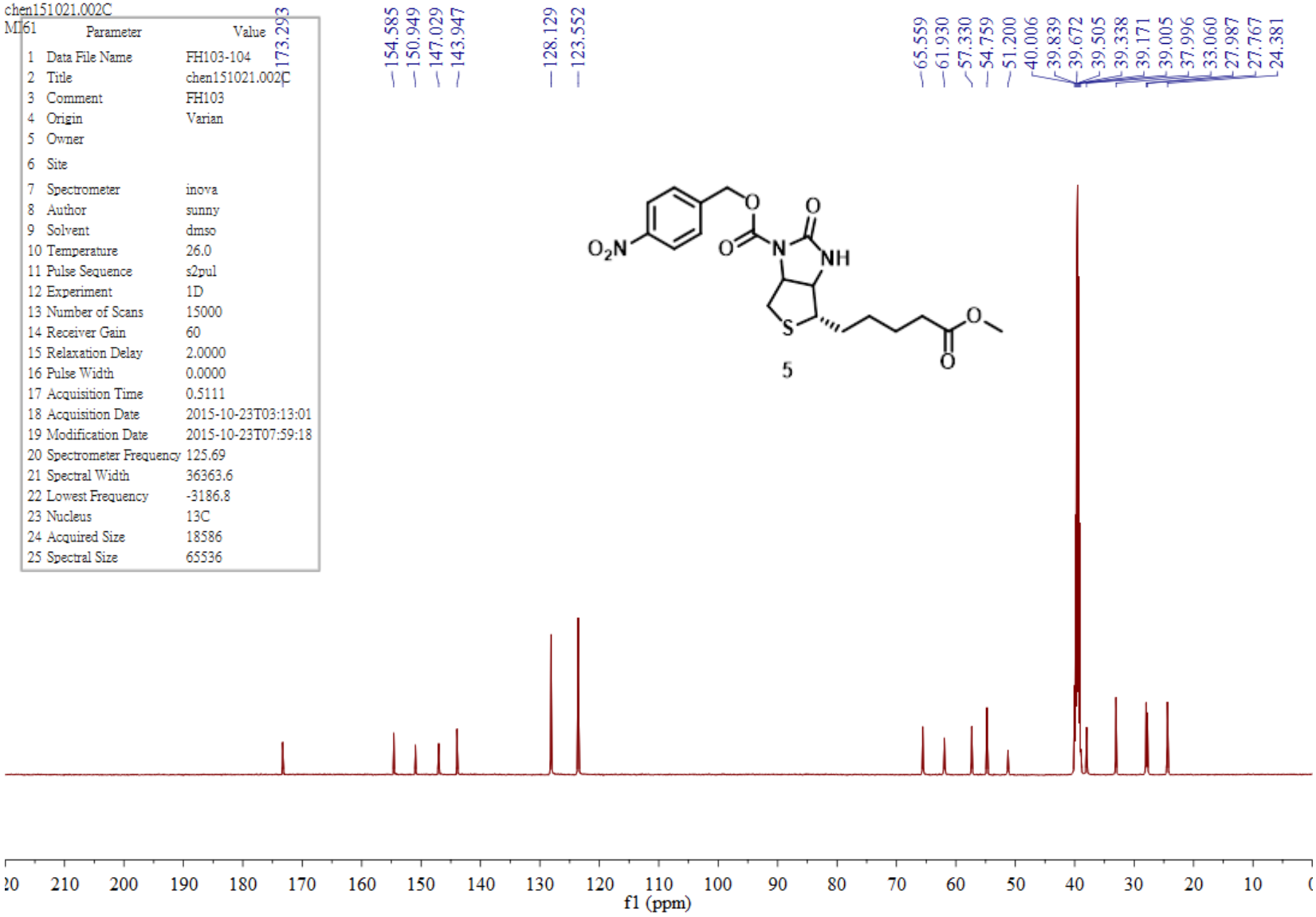
Deconvoluted MS

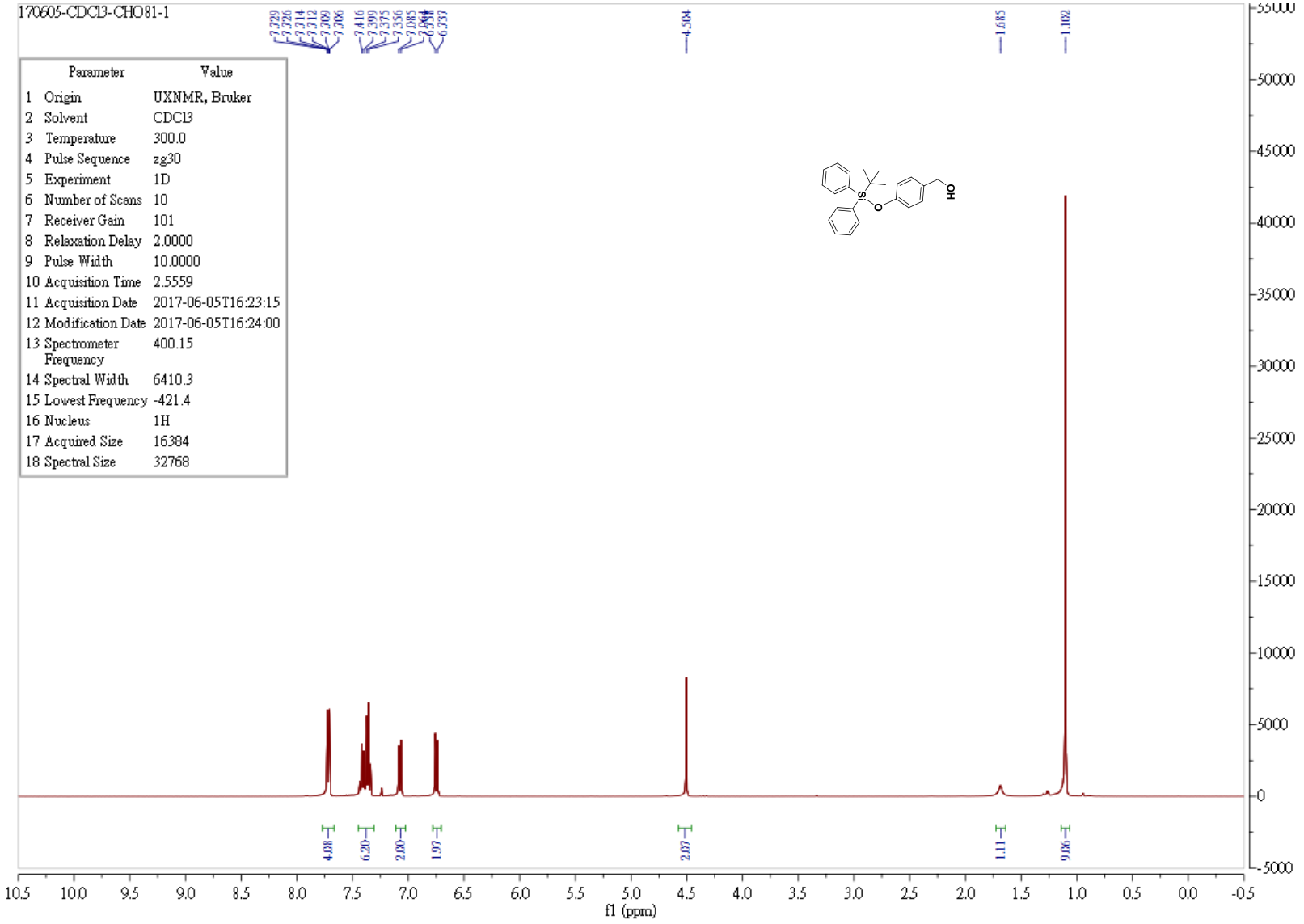
File: D:\2015-11\1103\SMS0053020151103_MI61(HR)_ESI.trans
Base-Peak Amplitude: 48.6073 Total Intensity: 391.211 Scans: 1 Positive Ions
Broadband ADC Rate: 2000000 ADC Gain: X1 Transient Points: 2048K

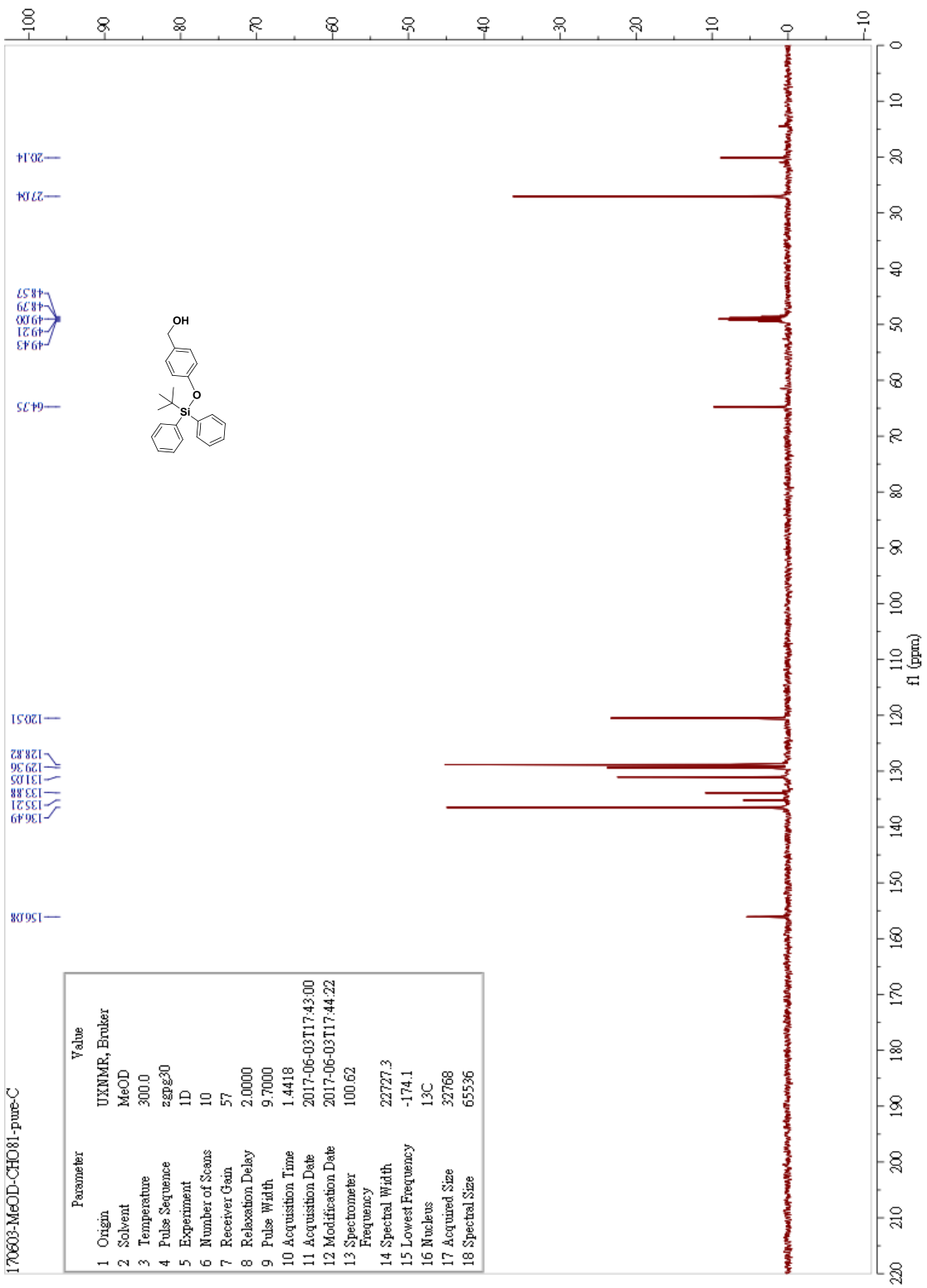
Remove Noises 03-NOV-2015 16:08:11

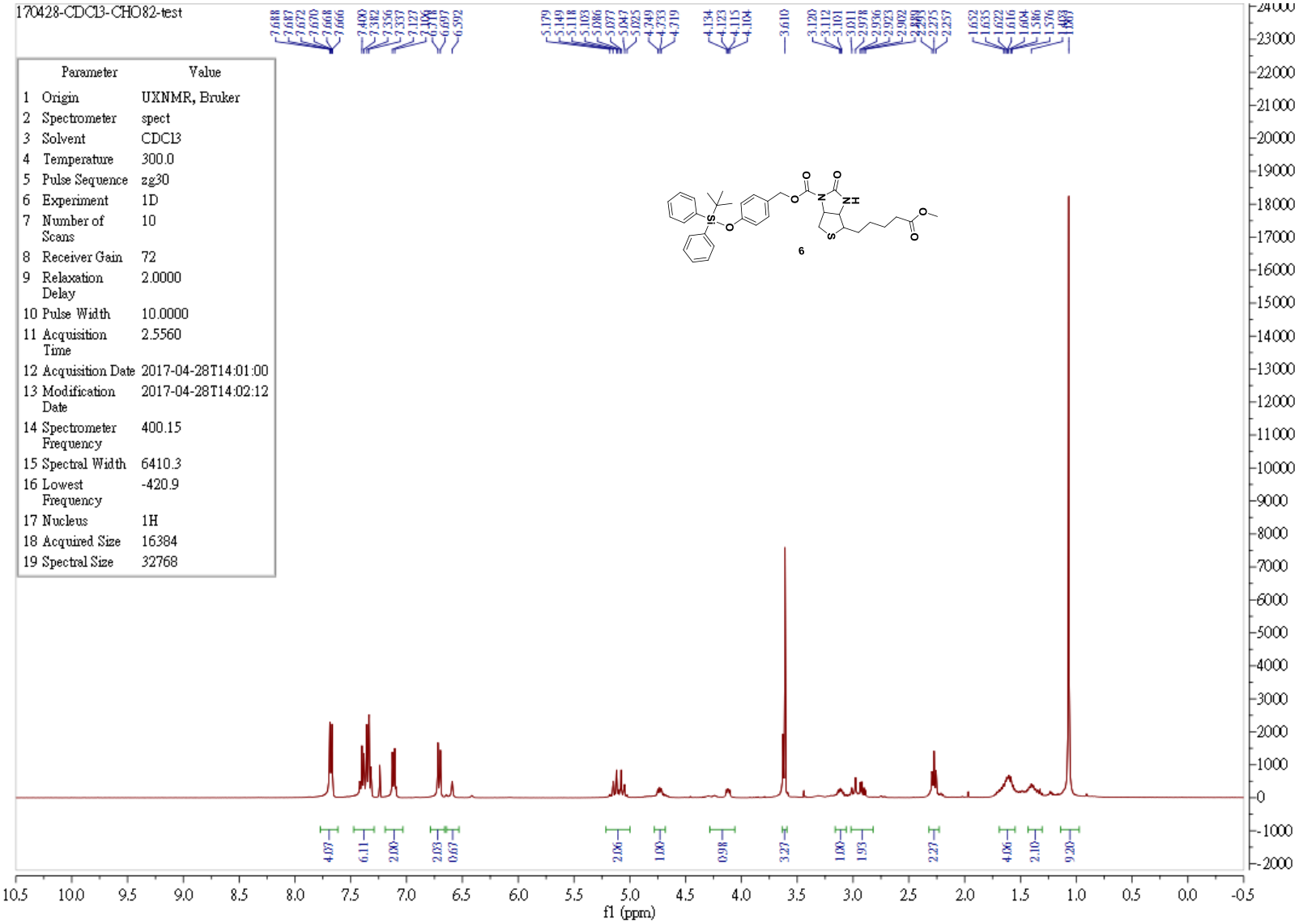


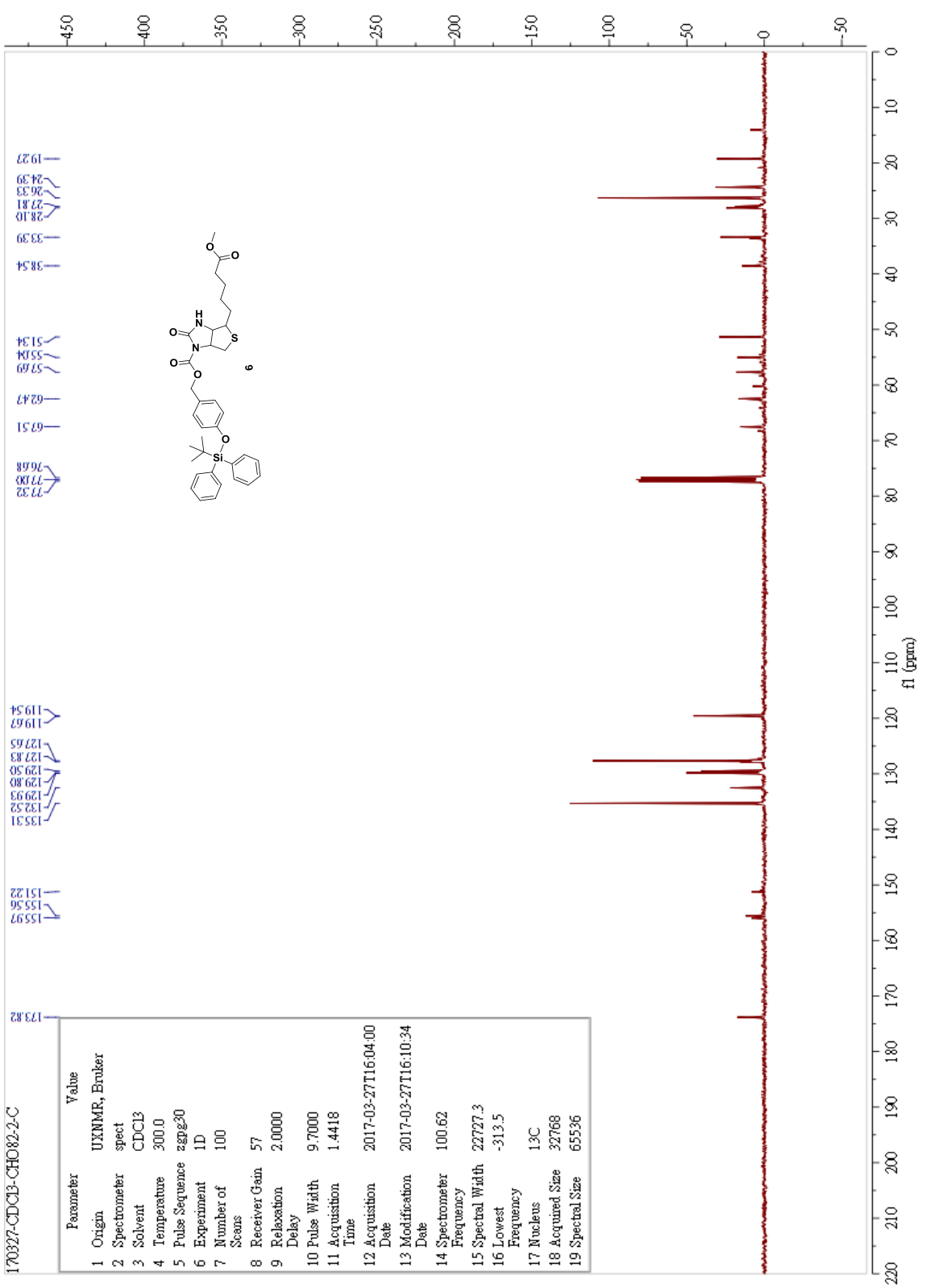

chen151021.002H
MI61
Parameter Value
1 Data File Name chen151021.002H.fid/ fid
2 Title chen151021.002H
3 Comment MI61
4 Origin Varian
5 Owner
6 Site
7 Spectrometer inova
8 Author sunny
9 Solvent dmso
10 Temperature 26.0
11 Pulse Sequence s2pul
12 Experiment 1D
13 Number of Scans 32
14 Receiver Gain 60
15 Relaxation Delay 1.0000
16 Pulse Width 0.0000
17 Acquisition Time 2.0436
18 Acquisition Date 2015-10-22T16:45:01
19 Modification Date 2015-10-22T16:45:42
20 Spectrometer Frequency 499.81
21 Spectral Width 9000.9
22 Lowest Frequency -1005.2
23 Nucleus 1H
24 Acquired Size 18394
25 Spectral Size 65536
8.243
8.226
7.772
7.696
7.679
5.330
4.790
4.779
4.765
4.156
4.147
4.141
4.132
3.570
3.347
3.340
3.189
2.985
2.975
2.958
2.494
2.490
2.487
2.295
2.280
1.829
1.610
1.563
1.549
1.534
1.521
1.471
1.454
1.349
1.335
1.318
1.304
O2N
O
N
NH
S
5
2.00
1.06
2.01
2.06
1.04
1.06
2.86
1.07
2.03
2.04
6.62
f1 (ppm)


chen151021.002C
M161
Parameter Value
1 Data File Name FH103-104
2 Title chen151021.002C
3 Comment FH103
4 Origin Varian
5 Owner
6 Site
7 Spectrometer inova
8 Author sunny
9 Solvent dmso
10 Temperature 26.0
11 Pulse Sequence s2pul
12 Experiment 1D
13 Number of Scans 15000
14 Receiver Gain 60
15 Relaxation Delay 2.0000
16 Pulse Width 0.0000
17 Acquisition Time 0.5111
18 Acquisition Date 2015-10-23T03:13:01
19 Modification Date 2015-10-23T07:59:18
20 Spectrometer Frequency 125.69
21 Spectral Width 36363.6
22 Lowest Frequency -3186.8
23 Nucleus 13C
24 Acquired Size 18586
25 Spectral Size 65536
173.293
154.585
150.949
147.029
143.947
128.129
123.552
65.559
61.930
57.330
54.759
51.200
40.006
39.839
39.672
39.505
39.338
39.171
39.005
37.996
33.060
27.987
27.767
24.381
O2N
O
N
NH
S
5
f1 (ppm)


170605-CDCl3-CHO81-1
7.729
7.726
7.714
7.712
7.709
7.706
7.416
7.399
7.375
7.356
7.085
6.959
6.737
4.504
1.685
1.102
Parameter Value
1 Origin UXNMR, Bruker
2 Solvent CDCl3
3 Temperature 300.0
4 Pulse Sequence zg30
5 Experiment 1D
6 Number of Scans 10
7 Receiver Gain 101
8 Relaxation Delay 2.0000
9 Pulse Width 10.0000
10 Acquisition Time 2.5559
11 Acquisition Date 2017-06-05T16:23:15
12 Modification Date 2017-06-05T16:24:00
13 Spectrometer Frequency 400.15
14 Spectral Width 6410.3
15 Lowest Frequency -421.4
16 Nucleus 1H
17 Acquired Size 16384
18 Spectral Size 32768
Si
O
OH
4.08
6.20
2.00
1.97
2.07
1.11
9.06
f1 (ppm)


170603-MeOD-CHO81-pure-C
Parameter Value
1 Origin UXNMR, Bruker
2 Solvent MeOD
3 Temperature 300.0
4 Pulse Sequence zgpg30
5 Experiment 1D
6 Number of Scans 10
7 Receiver Gain 57
8 Relaxation Delay 2.0000
9 Pulse Width 9.7000
10 Acquisition Time 1.4418
11 Acquisition Date 2017-06-03T17:43:00
12 Modification Date 2017-06-03T17:44:22
13 Spectrometer Frequency 100.62
14 Spectral Width 22727.3
15 Lowest Frequency -174.1
16 Nucleus 13C
17 Acquired Size 32768
18 Spectral Size 65536
156.98
136.49
135.21
133.88
131.05
129.36
128.82
120.51
64.75
49.43
49.21
48.99
48.79
48.57
27.04
20.14
OH
O
Si
f1 (ppm)

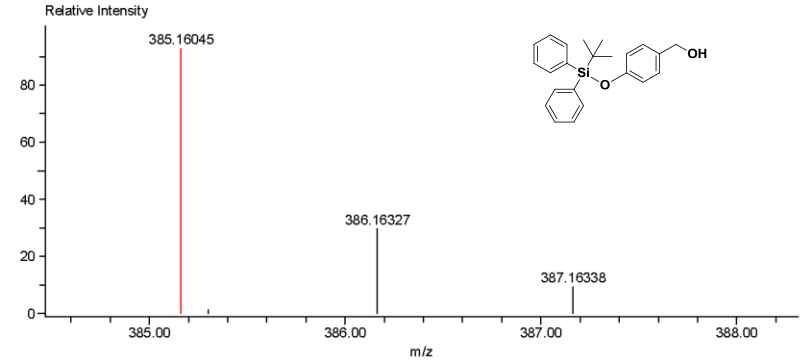

170428-CDCl3-CHO82-test
Parameter Value
1 Origin UXNMR, Bruker
2 Spectrometer spect
3 Solvent CDCl3
4 Temperature 300.0
5 Pulse Sequence zg30
6 Experiment 1D
7 Number of Scans 10
8 Receiver Gain 72
9 Relaxation Delay 2.0000
10 Pulse Width 10.0000
11 Acquisition Time 2.5560
12 Acquisition Date 2017-04-28T14:01:00
13 Modification Date 2017-04-28T14:02:12
14 Spectrometer Frequency 400.15
15 Spectral Width 6410.3
16 Lowest Frequency -420.9
17 Nucleus 1H
18 Acquired Size 16384
19 Spectral Size 32768
6
f1 (ppm)


170327-CDCl3-CHO82-2-C
Parameter Value
1 Origin UXNMR, Bruker
2 Spectrometer spect
3 Solvent CDCl3
4 Temperature 300.0
5 Pulse Sequence zgpg30
6 Experiment 1D
7 Number of Scans 100
8 Receiver Gain 57
9 Relaxation Delay 2.0000
10 Pulse Width 9.7000
11 Acquisition Time 1.4418
12 Acquisition Date 2017-03-27T16:04:00
13 Modification Date 2017-03-27T16:10:34
14 Spectrometer Frequency 100.62
15 Spectral Width 22727.3
16 Lowest Frequency -313.5
17 Nucleus 13C
18 Acquired Size 32768
19 Spectral Size 65536
f1 (ppm)

Data:CHO81
Comment:
Description:
Ionization Mode:ESI+
History:Average(MS[1] 0.62..0.72)

Acquired:6/23/2017 2:11:44 AM
Operator:AccuTOF
m/z Calibration File:20161229TFANa_...
Created:7/6/2017 8:47:05 PM
Created by:

Charge number:1 Tolerance:10.00[ppm], 5.00 .. 15.00[m... Unsaturation Number:-20.0 .. 100.0 (F...
Element:^{12}C:23 .. 23, ^{1}H:26 .. 26, ^{35}Cl:0 .. 0, ^{14}N:0 .. 0, ^{23}Na:1 .. 1, ^{16}O:2 .. 2, ^{32}S:0 .. 0, ^{28}Si:1 .. 1

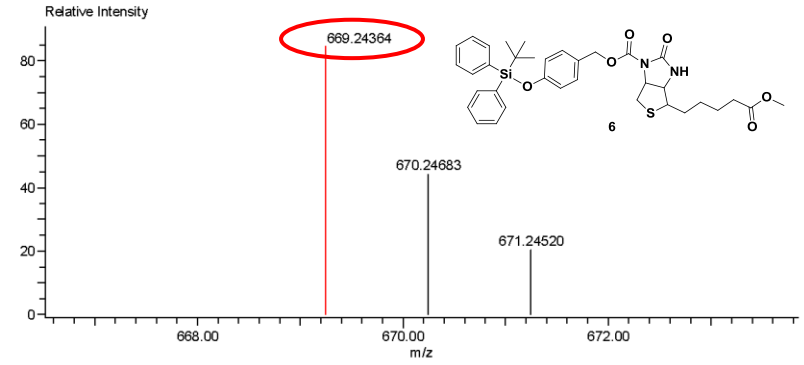


Mass	Intensity	Calc. Mass	Mass Difference [mDa]	Mass Difference [ppm]	Possible Formula
385.16045	31564.36	385.15998	0.47	1.23	$^{12}C_{23}{}^{1}H_{26}{}^{23}Na_{1}{}^{16}O_{2}{}^{28}Si_{1}$

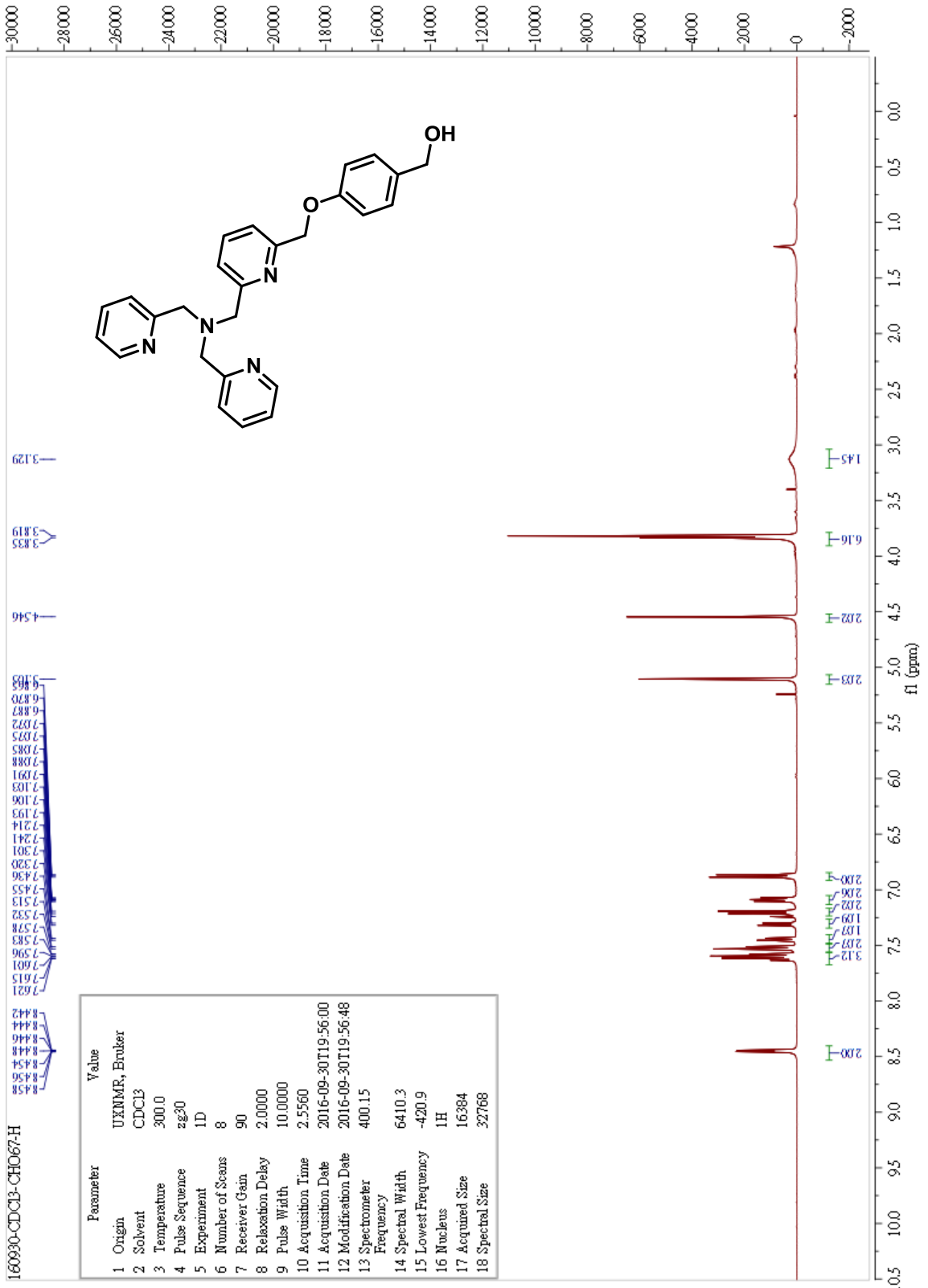
Data:CHO82
Comment:
Description:
Ionization Mode:ESI+
History:Average(MS[1] 0.80..0.87)

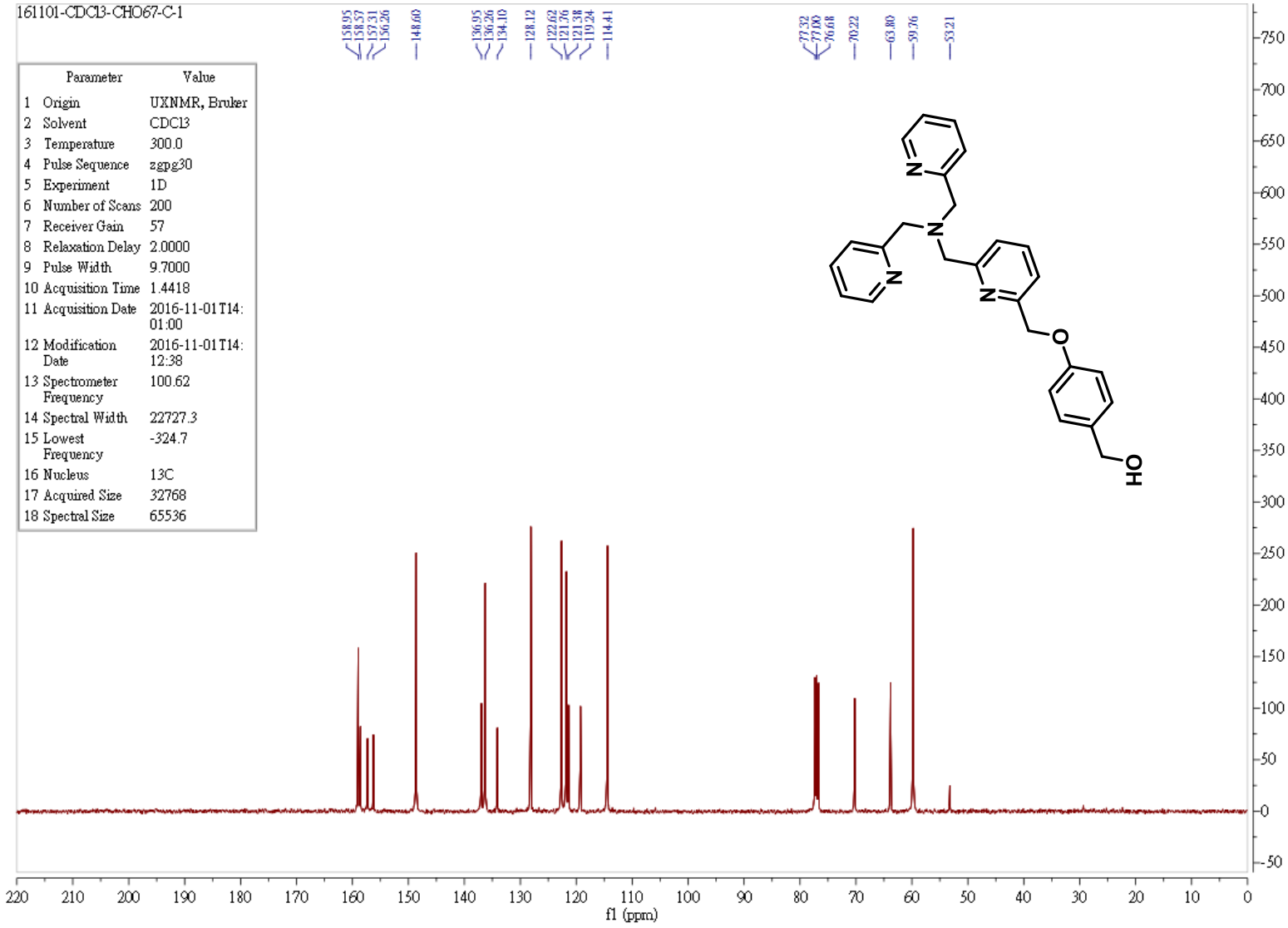
Acquired:6/9/2017 12:16:13 AM
Operator:AccuTOF
m/z Calibration File:20161229TFANa_...
Created:7/6/2017 8:26:20 PM
Created by:

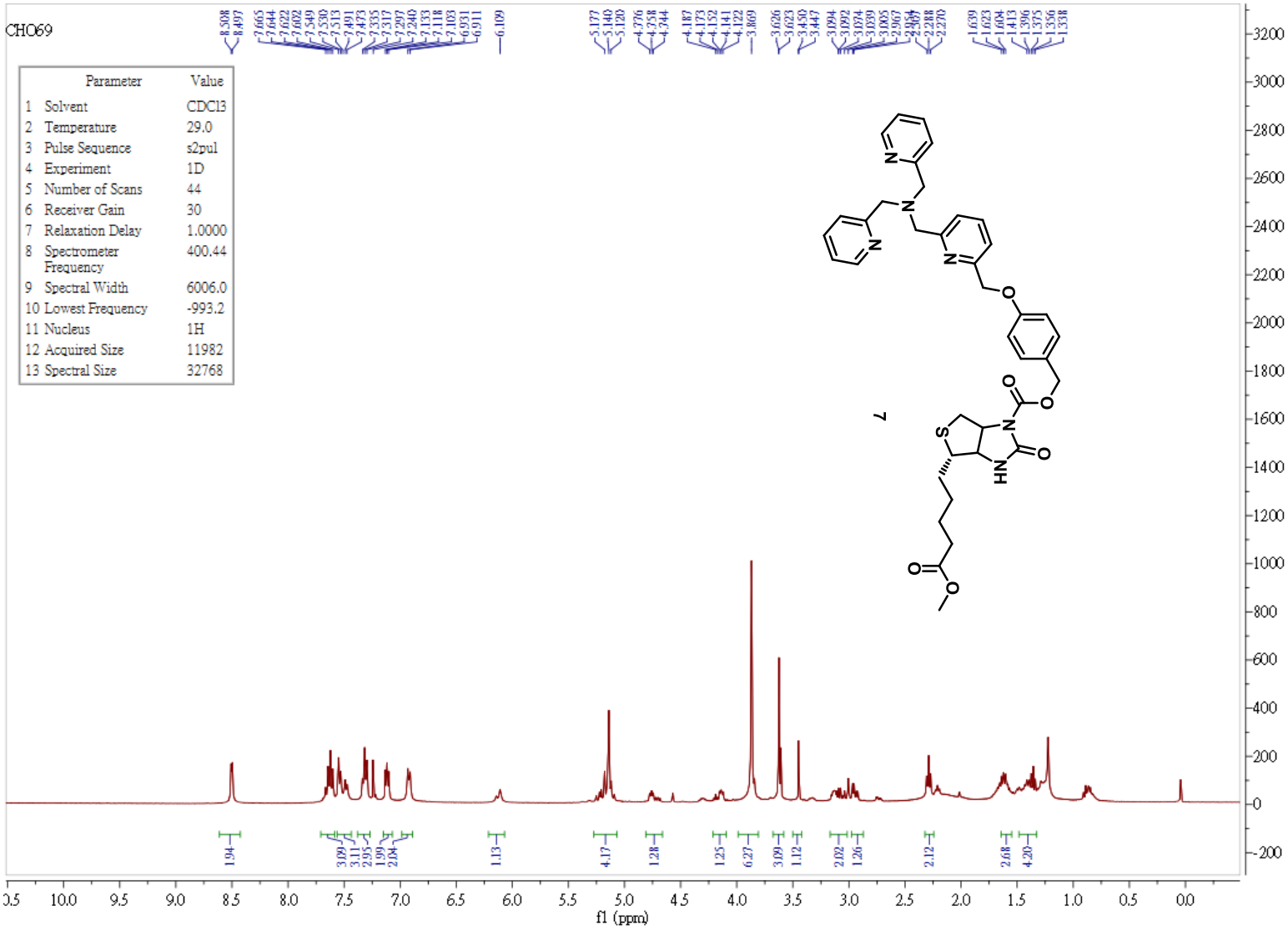
Charge number:1 Tolerance:10.00[ppm], 5.00 .. 15.00[m... Unsaturation Number:-20.0 .. 100.0 (F...
Element:^{12}C:35 .. 35, ^{1}H:42 .. 42, ^{35}Cl:0 .. 0, ^{14}N:2 .. 2, ^{23}Na:1 .. 1, ^{16}O:6 .. 6, ^{32}S:1 .. 1, ^{28}Si:1 .. 1

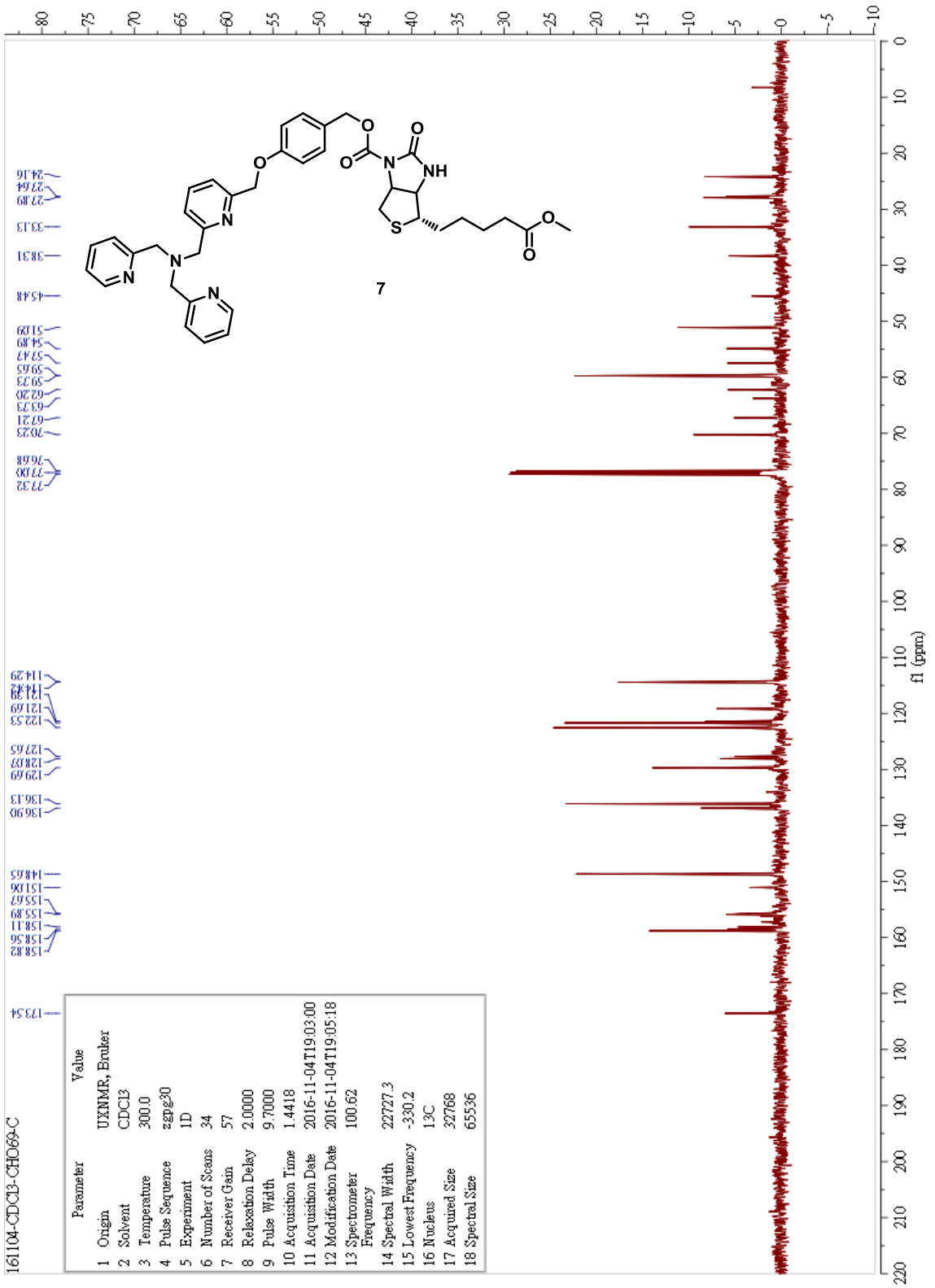


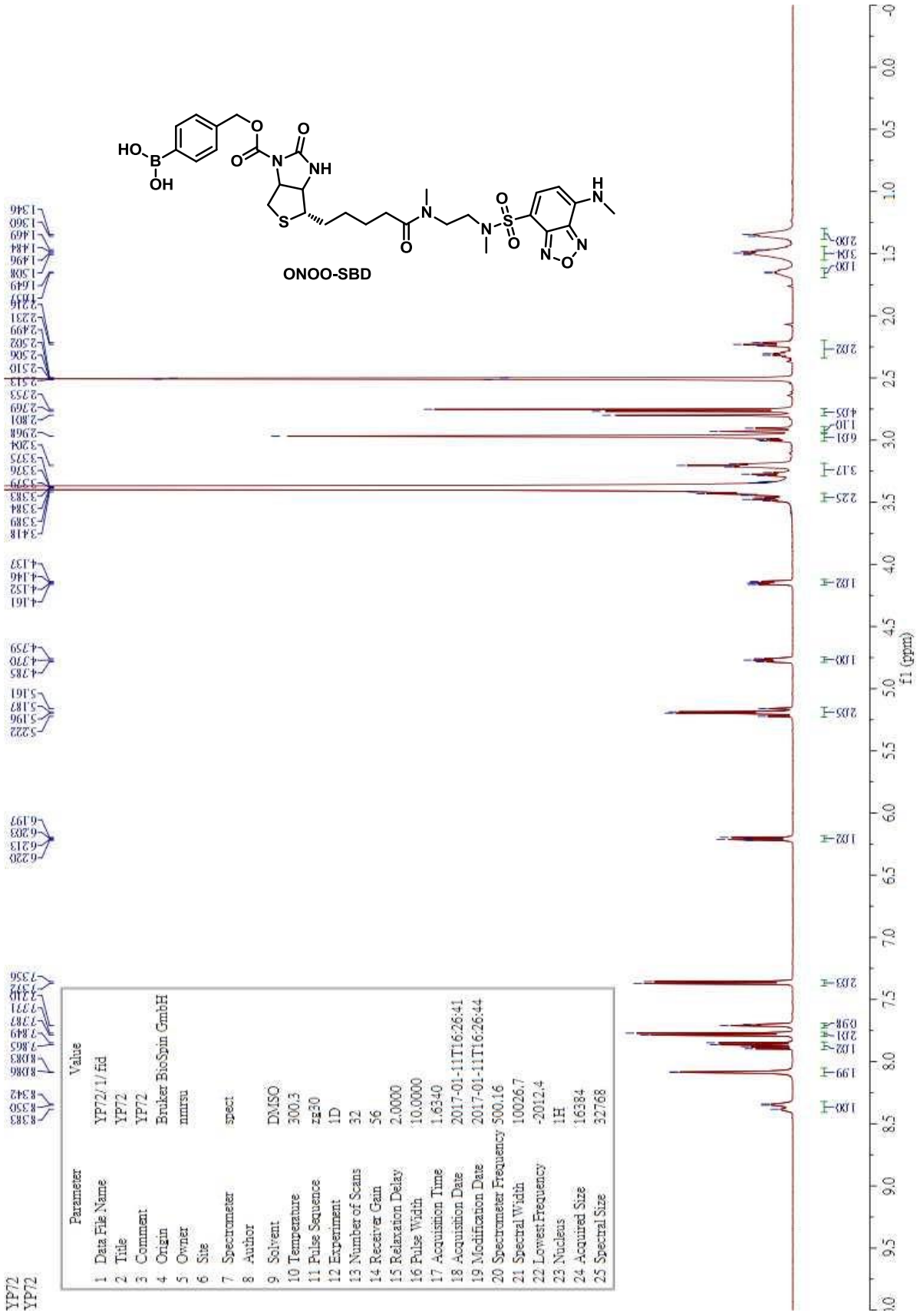
Mass	Intensity	Calc. Mass	Mass Difference [mDa]	Mass Difference [ppm]	Possible Formula
669.24364	12453.47	669.24305	0.58	0.87	$^{12}C_{35}{}^{1}H_{42}{}^{14}N_{2}{}^{23}Na_{1}{}^{16}O_{6}{}^{32}S_{1}{}^{28}Si_{1}$


160930-CDCl3-CHO67-H
Parameter Value
1 Origin UXNMR, Bruker
2 Solvent CDCl3
3 Temperature 300.0
4 Pulse Sequence zg30
5 Experiment 1D
6 Number of Scans 8
7 Receiver Gain 90
8 Relaxation Delay 2.0000
9 Pulse Width 10.0000
10 Acquisition Time 2.5560
11 Acquisition Date 2016-09-30T19:56:00
12 Modification Date 2016-09-30T19:56:48
13 Spectrometer Frequency 400.15
14 Spectral Width 6410.3
15 Lowest Frequency -420.9
16 Nuclens 1H
17 Acquired Size 16384
18 Spectral Size 32768
OH
O
N
N
N
f1 (ppm)

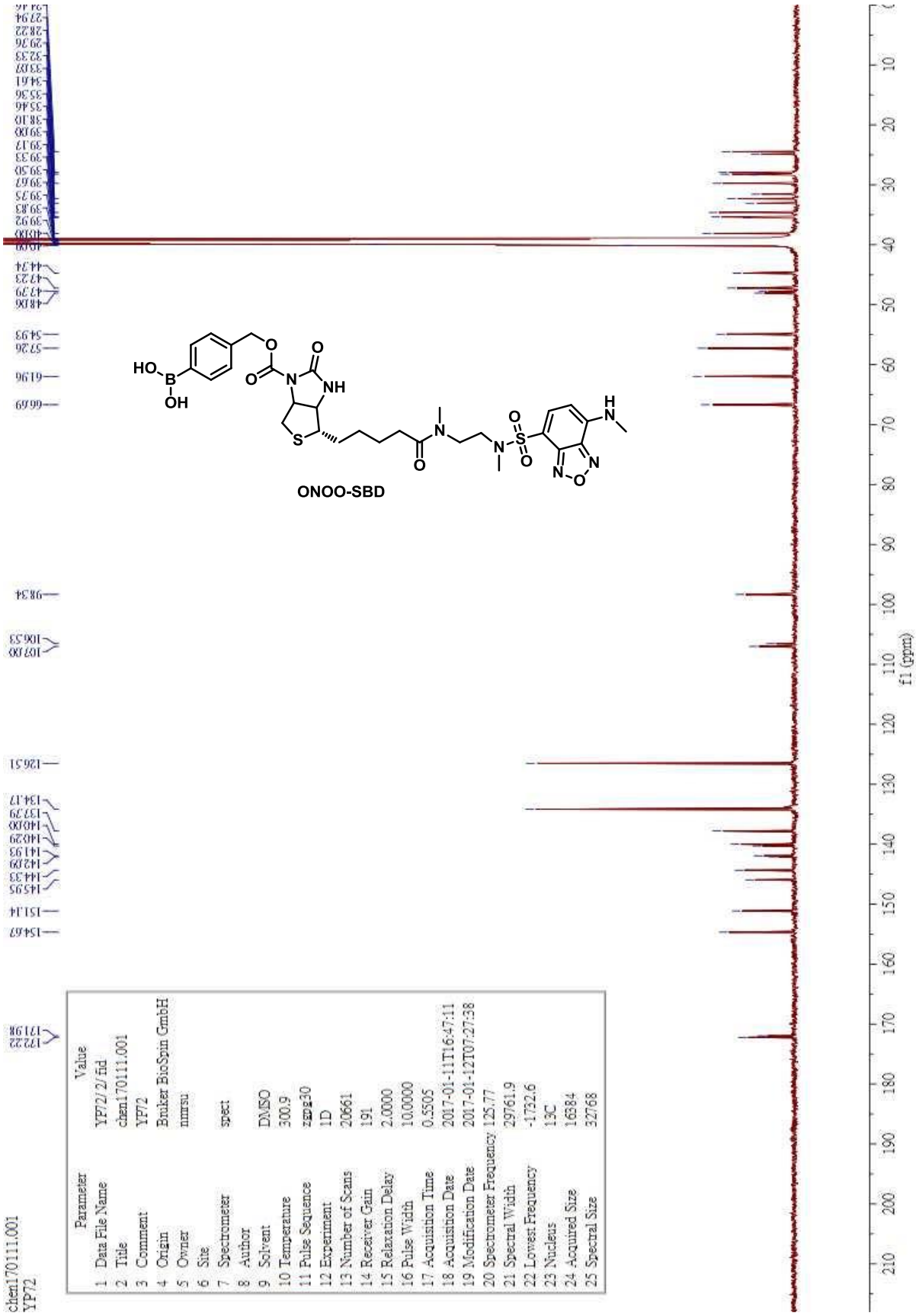

161101-CDCl3-CHO67-C-1
158.95
158.57
157.31
156.26
148.60
136.95
136.26
134.10
128.12
122.62
121.76
121.38
119.24
114.41
77.32
77.00
76.68
70.22
63.80
59.76
53.21
Parameter Value
1 Origin UXNMR, Bruker
2 Solvent CDCl3
3 Temperature 300.0
4 Pulse Sequence zgpg30
5 Experiment 1D
6 Number of Scans 200
7 Receiver Gain 57
8 Relaxation Delay 2.0000
9 Pulse Width 9.7000
10 Acquisition Time 1.4418
11 Acquisition Date 2016-11-01T14:01:00
12 Modification Date 2016-11-01T14:12:38
13 Spectrometer Frequency 100.62
14 Spectral Width 22727.3
15 Lowest Frequency -324.7
16 Nucleus 13C
17 Acquired Size 32768
18 Spectral Size 65536
N
N
N
O
HO
f1 (ppm)


CHO69
Parameter Value
1 Solvent CDCl3
2 Temperature 29.0
3 Pulse Sequence s2pul
4 Experiment 1D
5 Number of Scans 44
6 Receiver Gain 30
7 Relaxation Delay 1.0000
8 Spectrometer Frequency 400.44
9 Spectral Width 6006.0
10 Lowest Frequency -993.2
11 Nucleus 1H
12 Acquired Size 11982
13 Spectral Size 32768
7
f1 (ppm)


161104-CDCl3-CHO69-C
Parameter Value
1 Origin UXNMR, Bruker
2 Solvent CDCl3
3 Temperature 300.0
4 Pulse Sequence zgpg30
5 Experiment 1D
6 Number of Scans 34
7 Receiver Gain 57
8 Relaxation Delay 2.0000
9 Pulse Width 9.7000
10 Acquisition Time 1.4418
11 Acquisition Date 2016-11-04T19:03:00
12 Modification Date 2016-11-04T19:05:18
13 Spectrometer Frequency 100.62
14 Spectral Width 22727.3
15 Lowest Frequency -330.2
16 Nucleus 13C
17 Acquired Size 32768
18 Spectral Size 65536
7
f1 (ppm)



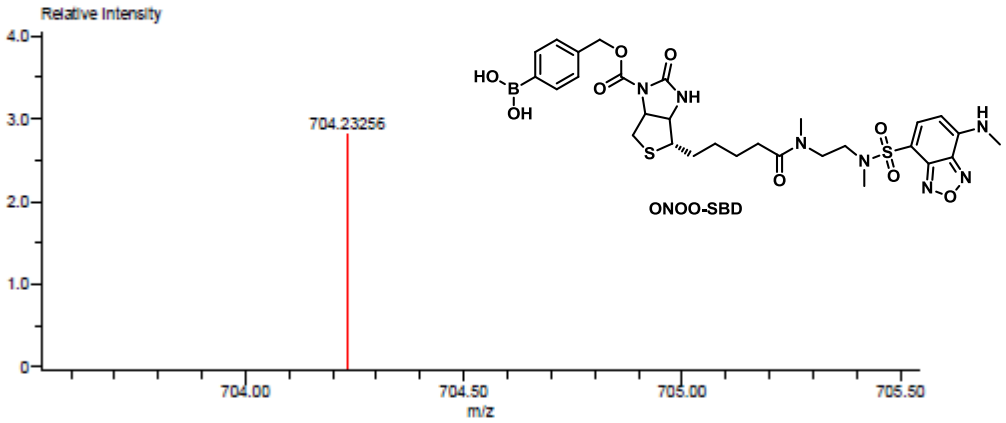
	Parameter	Value
1	Data File Name	YP72/1/fid
2	Title	YP72
3	Comment	YP72
4	Origin	Bruker BioSpin GmbH
5	Owner	nmrsu
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	DMSO
10	Temperature	300.3
11	Pulse Sequence	zg30
12	Experiment	1D
13	Number of Scans	32
14	Receiver Gain	56
15	Relaxation Delay	2.0000
16	Pulse Width	10.0000
17	Acquisition Time	1.6340
18	Acquisition Date	2017-01-11T16:26:41
19	Modification Date	2017-01-11T16:26:44
20	Spectrometer Frequency	500.16
21	Spectral Width	10026.7
22	Lowest Frequency	-2012.4
23	Nucleus	1H
24	Acquired Size	16384
25	Spectral Size	32768


ONOO-SBD
f1 (ppm)
chen170111.001
YP72
Parameter Value
1 Data File Name YP72/2/fid
2 Title chen170111.001
3 Comment YP72
4 Origin Bruker BioSpin GmbH
5 Owner nmrsu
6 Site
7 Spectrometer spect
8 Author
9 Solvent DMSO
10 Temperature 300.9
11 Pulse Sequence zgpg30
12 Experiment 1D
13 Number of Scans 20661
14 Receiver Gain 191
15 Relaxation Delay 2.0000
16 Pulse Width 10.0000
17 Acquisition Time 0.5505
18 Acquisition Date 2017-01-11T16:47:11
19 Modification Date 2017-01-12T07:27:38
20 Spectrometer Frequency 125.77
21 Spectral Width 29761.9
22 Lowest Frequency -1732.6
23 Nucleus 13C
24 Acquired Size 16384
25 Spectral Size 32768

Data:pos TK06
Comment:
Description:
Ionization Mode:ESI+
History:Average(MS[1] 0.56..0.60)

Acquired:11/1/2017 3:45:07 PM
Operator:AccuTOF
m/z Calibration File:20161229TFANa_...
Created:11/1/2017 5:12:05 PM
Created by:AccuTOF

Charge number:1 Tolerance:10.00[ppm], 5.00 .. 15.00[m... Unsaturation Number:-20.0 .. 100.0 (F...
Element:^{12}C:29 .. 29, ^{1}H:39 .. 39, ^{11}B:1 .. 1, ^{14}N:7 .. 7, ^{16}O:9 .. 9, ^{32}S:2 .. 2



Mass	Intensity	Calc. Mass	Mass Difference [mDa]	Mass Difference [ppm]	Possible Formula
704.23256	1185.21	704.23437	-1.82	-2.58	$^{12}C_{29}{}^{1}H_{39}{}^{11}B_{1}{}^{14}N_{7}{}^{16}O_{9}{}^{32}S_{2}$