Electronic Supplementary Information
for
Boronic Acid-modified DNA that Changes Fluorescent Properties upon Carbohydrate Binding †

Xiaochuan Yang, Chaofeng Dai, Angie Dayan Calderon Molina, and Binghe Wang*

Experimental Section

General: Unless otherwise noted, starting materials and solvents were purchased from Aldrich and Acros, and used without purification. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker 400 MHz spectrophotometer in a deuterated solvent with TMS (δ = 0.00 ppm) or NMR solvent as the internal reference unless otherwise specified. ^{31}P NMR was recorded using 85% H_3PO_4 as an external reference (in a insert). HPLC purification was carried out with a Zobax C18 reversed-phase column (9.4 mm×25 cm). Fluorescence spectra were recorded on a Shimadzu RF-5301 PC spectrofluorometer. Absorption spectra were recorded on a Shimadzu UV-1700 UV/Vis spectrophotometer. Quartz cuvettes were used in all fluorescence and UV studies. All pH values were determined by a UB-10 Ultra basic benchtopp H meter (Denver Instruments). For all reactions, analytical grade solvents were used. Anhydrous solvents were used for all moisture-sensitive reactions. Mass spectra were recorded on a Waters Micromass LC-Q-TOF micro spectrometer or an ABI4800 MALDI-TOF-TOF mass spectrometer at the Georgia State University Mass Spectrometry Facilities.

1. Synthesis of Naphthalimide-based Long-wavelength Boronic Acid Modified TTP (NB-TTP):

The first three steps were carried out following published procedures.[1,2]
Preparation of 4-aminomethylbenzyl alcohol:
4-(Hydroxymethyl)benzonitrile (1.0 g, 7.5 mmol) dissolved in dry THF (20 ml) was added slowly into a suspension of $LiAlH_4$ (866 mg, 22.8 mmol) in THF(20 ml) at RT. Foaming with bubble formation was observed while stirring. The light yellow-green suspension was refluxed under N_2 overnight. After cooling down to RT, MeOH (5 ml) was added to the suspension to quench the reaction. The suspension generated foams while being stirred and cooled in an ice-bath. When no more bubble formation was observed, NaOH (20%, 15 ml) solution was added into the suspension. The solution became colorless while white precipitate was observed. The solution was concentrated *in vaccuo* and the residue was suction filtered. The solid was repeatedly washed with CH_2Cl_2. The filtrate was combined and washed with H_2O. The organic phase was separated and concentrated on a rotavap. White solid was obtained as product (1.0 g, 98% yield). The product underwent prolonged drying *in vaccuo* to remove residue solvent before the next step reaction.
^{1}H-NMR ($CDCl_3$): δ 7.31 (m, 4H), 4.67(s, 2H), 3.85(s, 2H), 1.76 (s broad, 3H)
^{13}C-NMR ($CDCl_3$): δ 142.7, 139.9, 127.5, 127.5, 65.2, 46.4

Preparation of 4-boc-aminomethylbenzyl alcohol:
Into the solution of 4-aminomethylbenzyl alcohol (444 mg, 3.2 mmol) and triethylamine (0.47 ml, 3.3 mmol) in dry THF (5 ml) was added di-*t*-butyl dicarbonate (750 mg, 3.4 mmol) in dry THF (3 ml) dropwise. The mixture was stirred at RT overnight and then concentrated on a rotavap. The residue was dissolved in ethyl acetate (20 ml). The solution was sequentially washed with $NaHSO_4$ solution (5%, × 1), saturated $NaHCO_3$ solution (× 1) and brine (× 1), and then dried over $MgSO_4$. Filtration and solvent evaporation yielded a white powder product (720 mg, 94% yield).
^{1}H-NMR ($CDCl_3$): δ 7.27 (m, 4H), 4.92 (s broad, 1H), 4.65 (s, 2H), 4.27 (d, 2H), 2.22 (s broad, 1H), 1.45 (s, 9H)
^{13}C-NMR ($CDCl_3$): δ 156.1, 140.3, 138.4, 127.8, 127.4, 79.7, 65.1, 44.6, 28.6

Preparation of (4-boc-aminomethylbenzyl) methansulfonate (**2**):
Into the solution of 4-Boc-aminomethylbenzyl alcohol (700 mg, 3.0 mmol) and triethylamine (0.86 ml, 6.2 mmol) in dry THF (12 ml) in an ice bath, MsCl (0.38 ml, 4.9 mmol) was added dropwise. The mixture was stirred for 1 h in an ice bath. The solution was concentrated on a rotavap and the residue was dissolved in 25ml of ethyl acetate. The solution was sequentially washed with $NaHSO_4$ solution (5%, × 1), saturated $NaHCO_3$ solution (× 1) and brine (× 1), and then dried over $MgSO_4$. After filtration and solvent evaporation, a colorless oil product was obtained (896 mg, 96% yield), which turned into white crystal after prolonged drying *in vaccuo*.
^{1}H-NMR ($CDCl_3$): δ 7.35 (m, 4H), 5.22 (s, 2H), 4.95 (s broad, 1H), 4.33 (d, 2H), 2.91 (s, 3H), 1.46 (s, 9H)
^{13}C-NMR ($CDCl_3$): δ 156.1, 140.7, 132.5, 129.4 128.0, 79.9 71.4, 44.4, 38.5, 28.6

Preparation of 4-amino-*N*-(4'-boc-aminomethylbenzyl)naphthalimide (**3**):
4-Amino-naphthalimide (820 mg, 3.85 mmol) in dry DMF (20 ml) was treated with 2 M NaOMe in MeOH (3 ml) until it became a homogenous solution with a deep red color (took several minutes). (4-Boc-aminomethylbenzyl) methansulfonate (1.21 g, 3.85 mmol) was then added. The solution was stirred for 3 h and then worked up by adding H_2O (100 ml). The suspension was extracted with ethyl acetate (100 ml, × 2). The organic extractions were combined and concentrated into 100 ml. The solution was washed with H_2O (25 ml, × 3) and dried over $MgSO_4$. The removal of solvent on a rotavap gave an orange powder product (1.5 g, 90% yield).
^{1}H-NMR (D_6-DMSO): δ 8.63 (d, 1H, J = 7.6 Hz), 8.44 (dd, 1H, J_1 = 7.2 Hz, J_2 = 0.8 Hz), 8.21 (d, 1H, J = 9.2 Hz), 7.66 (t, 1H, J = 8.0 Hz,), 7.49 (s broad, 2H), 7.20 (m, 4H), 6.86 (d, 1H, J = 8.4 Hz), 5.19 (s, 2H), 4.06 (d, 2H, J = 6 Hz), 1.366 (s, 9H)
^{13}C-NMR (D_6-DMSO): δ 163.8, 162.9, 155.7, 152.9, 138.8, 136.4, 134.2, 131.2, 129.8, 129.5, 127.4, 126.9, 124.0, 121.7, 119.4, 108.2, 107.3, 77.7, 43.1, 42.2, 28.2.
MS (ESI-): *m/z* (%) = 430.3 (100) $[M-H]^-$.
Exact mass: $C_{25}H_{24}N_3O_4$ calc. 430.1767; found 430.1778

Preparation o f4-(2-bromobenzyl)amino-*N*-(4'-boc-aminomethylbenzyl)naphthalimide (**4**):
4-Amino-*N*-(4'-Boc-aminomethylbenzyl)naphthalimide (**3**) (1.50 g, 3.49 mmol) and sodium hydride (60% dispersed in mineral oil, 306 mg, 7.66 mmol) were mixed in dry DMF (20 ml). The mixture was stirred for 5 min and then 2-bromobenzyl bromide (870 mg, 3.49 mmol) was added. The mixture was stirred for 3 h before quenching with water (150 ml). The suspension was extracted with ethyl acetate (25 ml, × 3). The organic extractions were combined and washed with water (50 ml). After separation, the organic solvent was removed on a rotavap and the residue was purified on a silica gel column (ethyl acetate/hexane, 1:4). A yellow powder product (840 mg, 40% yield) was obtained.
^{1}H-NMR (D_6-DMSO): δ 8.58 (d, 1H, J = 7.2 Hz), 8.43 (d, 1H, J = 8.4 Hz), 8.13 (d, 1H, J = 8.4 Hz), 7.64~7.61 (m, 2H), 7.49 (d, 2H, J = 8.0 Hz), 7.37 (dd, 1H, J_1 = 7.6 Hz, J_2 = 1.6 Hz), 7.30~7.28 (m, 1H), 7.27~7.18 (m, 2H), 6.68 (d, 1H, J = 8.4 Hz), 5.78 (s, 1H), 5.33 (s, 2H), 4.75 (s, 1H), 4.69 (d, 2H, J = 5.6 Hz), 4.24 (d, 2H, J = 5.6 Hz), 1.43 (s, 9H)
^{13}C-NMR (D_6-DMSO): δ 164.8, 164.2, 156.0, 149.1, 138.0, 137.2, 136.1, 134.7, 133.5, 131.6, 130.0, 129.8, 129.5, 129.3, 128.1, 127.8, 126.2, 125.2, 123.8, 123.4, 120.6, 111.3, 105.3, 79.6, 48.3, 44.7, 43.2, 28.6
MS (ESI+): *m/z* (%) = 600.5 (100) $[M+H]^+$.
Exact mass: $C_{32}H_{31}BrN_3O_4$ calc. 600.1498; found 600.1504

Preparation of 4-(2-dihydroxylboryl-benzyl)amino-*N*-(4'-boc-aminomethylbenzyl)naphthalimide (**5**):
4-(2-Bromobenzyl)amino-*N*-(4'-boc-aminomethylbenzyl)naphthalimide (**4**) (391 mg, 0.65 mmol), $PdCl_2$(dppf) (160 mg, 0.20 mmol), bis(neopentyl glycolato)diboron (368 mg, 1.63 mmol) and KOAc (198 mg, 2.02 mmol) were mixed in a dry flask under N_2. Anhydrous DMSO (5 ml) was injected into the mixture. The solution was heated at 80~85℃ under N_2 for 5 h. Then the solution was cooled down to RT. Water (25 ml) was then added to quench the reaction. The solution was extracted with

ethyl acetate (25ml, × 2). The extractions were combined, washed with water (25ml, × 2) and concentrated on a rotavap. The residue was purified on a silica gel column, and the crude product was eluted out by ethyl acetate/MeOH = 10:1. The crude product was further purified on two 20 × 20 cm preparatory TLC plates (ethyl acetate/MeOH = 5:1). The orange fluorescent band (R_f = 0.1~0.5) was cut out and extracted with MeOH. Orange color solid product was obtained (167 mg, 45% yield)
^{1}H-NMR (MeOD): δ 8.22 (d, 1H, *J* = 8.0 Hz), 8.04 (d, 1H, *J* = 8.8 Hz), 7.58 (d, 1H, *J* = 7.2 Hz), 7.42 (d, 2H, *J* = 7.6 Hz), 7.33~7.25 (m, 4H), 7.19~7.16 (m, 3H), 6.17 (d, 1H, *J* = 8.4 Hz), 5.30 (s, 2H), 4.55 (s, 2H), 4.15 (s, 2H), 1.42 (s, 9H)
^{13}C-NMR (MeOD): δ 167.8, 166.2, 164.7, 158.7, 141.6, 139.5, 138.9, 133.6, 130.9, 129.4, 129.1, 129.0, 128.8, 128.3, 127.5, 126.4, 119.6, 104.0, 101.8, 80.3, 47.7, 45.0, 43.8, 30.9, 28.9
MS (ESI-): *m/z* (%) = 564.3 (100) $[M-H]^-$.
Exact mass: $C_{32}H_{31}BN_3O_6$ calc. 564.2306; found 564.2310

Preparation of 4-(2-dihydroxylboryl-benzyl)amino-*N*-(4'-aminomethylbenzyl)naphthalimide (**6**):
4-(2-Dihydroxylboryl-benzyl)amino-*N*-(4'-Boc-aminomethylbenzyl)naphthalimide (**5**) (390 mg, 0.69 mmol) was suspended in CH_2Cl_2 (12 ml) and TFA (1.2 ml, 15.6 mmol) was added in one-shot. The mixture was stirred at RT for 0.5 h and then concentrated o rotavap. The residue was washed with a saturated K_2CO_3 solution. Then the residue was dissolved in methanol and loaded on two 20 × 20 cm preparatory TLC plates. 100% ethyl acetate was used to develop first. Later, ethyl acetate/MeOH (5:1) was used to develop the plate. The orange fluorescent band (R_f = 0.2~0.3) was cut out and extracted with MeOH. An orange color solid product was obtained (210 mg, 66% yield).
^{1}H-NMR (MeOD): δ 8.21 (d, 1H, *J* = 6.8 Hz), 8.03 (t, 1H, *J* = 4 Hz), 7.58 (d, 1H, *J* = 7.2 Hz), 7.44~7.37 (m, 4H), 7.29~7.25 (m, 4H), 7.17 (t, 1H, *J* = 7.2 Hz), 6.16 (d, 1H, *J* = 8.8 Hz), 5.32 (s, 2H), 4.55 (s, 2H), 3.86 (s, 2H),
^{13}C-NMR (MeOD): δ 167.8, 166.1, 141.6, 140.2, 138.8, 133.6, 130.9, 129.4, 129.3, 129.2, 128.9, 127.5, 126.3, 119.4, 103.8, 101.8, 47.9, 45.5, 43.8
MS (ESI-): *m/z* (%) = 492.3 (100) $[M-H-2H_2O+2MeOH]^-$, 478.3 (70) $[M-H-H_2O+MeOH]^-$,464.3 (50) $[M-H]^-$.
Exact mass: $C_{27}H_{23}BN_3O_4$ calc. 464.1782; found 464.1796

Preparation of 4-(2-dihydroxylboryl-benzyl)amino-*N*-(4'-azidoacetyl-aminomethylbenzyl)naphthalimide (**7**):
4-(2-Dihydroxylboryl-benzyl)amino-*N*-(4'-aminomethylbenzyl)naphthalimide (**6**) (210 mg, 0.45 mmol), EDCI (170 mg, 0.90 mmol), and azidoacetic acid (91 mg, 0.90 mmol) were mixed in anhydrous DMF (10 ml). The mixture was stirred at RT under N_2 for 1 h. The solution was concentrated by oil pump at 35 °C. The residue was rinsed with H_2O and then saturated K_2CO_3 solution. After that, the residue dissolved in ethanol was loaded on two 20 × 20 cm preparatory TLC plates and 100% ethyl acetate was used to develop first. Later, ethyl acetate/MeOH (10:1) was used to develop the plate. The orange fluorescent band (R_f = 0.2~0.3) was cut out and extracted with MeOH. An orange color solid product was obtained (241 mg, 98% yield).
^{1}H-NMR (MeOD): δ 8.23 (d, 1H, *J* = 7.2 Hz), 8.06 (d, 1H, *J* = 8.4 Hz), 7.59 (d, 1H, *J* = 6.8 Hz), 7.42 (d, 2H, *J* = 7.6 Hz), 7.33 (d, 2H, *J* = 8.0 Hz), 7.27 (t, 2H, *J* = 7.6 Hz), 7.21~7.17 (m, 3H), 6.19 (d, 1H, *J* = 8.8 Hz), 5.30 (s, 2H), 4.55 (s, 2H), 4.34 (s, 2H), 3.88 (s, 2H)
^{13}C-NMR (MeOD): δ 170.1, 167.8, 164.6, 141.5, 139.3, 138.9, 138.2, 133.6, 130.9, 129.4, 129.2, 128.9, 128.8, 127.6, 126.5, 119.8, 104.3, 101.8, 53.1, 47.5, 44.2, 44.0, 43.8, 30.9, 18.5
MS (ESI-): *m/z* (%) = 547.3 (100) $[M-H]^-$, 561.3 (50) $[M-H-H_2O+MeOH]^-$.
Exact mass: $C_{29}H_{24}BN_6O_5$ calc. 464.1782; found 464.1796

Preparation of NB-TTP:
4-(2-dihydroxylboryl-benzyl)amino-*N*-(4'-azidoacetyl-aminomethylbenzyl)naphthalimide (**7**) (10 mg, 0.018 mmol) and M-TTP (10 mg, 0.017 mmol) were mixed in a flask under N_2. MeOH/H_2O (1:1, 200 μl) was injected into the flask later to suspend the mixture with stirring. TBTA (1.7 mg, 0.0033 mmol)

and CuBr solution (0.24 mg, 0.0017 mmol) in 50 µl DMF were added later. The reaction mixture was sonicated for several minutes and then stirred at RT under N_2 for 5 h. The mixture was diluted by adding 1 ml of H_2O and then centrifuged at 13.5 krpm for 15 min. The aqueous supernatant was collected and the residue was repeatedly extracted with 100 mM NH NH_4HCO_3 buffer pH 8.0 until the extraction was no longer deep orange. The aqueous extractions were combined and purified by HPLC (C18 RP preparatory column). Elution condition: 100 mM NH_4HCO_3 buffer pH 8.0 /CH_3CN (3 ml/min), 0–10 min (CH_3CN 10%), 10–30 min (CH_3CN, 10-100%), 30–40 min (CH_3CN, 100%), 40–45 min (CH_3CN, 100-10%), 45–55 min (CH_3CN, 10%). Rt = 17.5-22.5 min. The collected fraction was lyophilized into orange color powder. The product was washed with MeOH and ice-cold water to remove residual starting material. Further purification of NB-TTP could be achieved by MeOH precipitation of the saturated aqueous solution. A bright red-orange colored powder was obtained as pure product (5 mg, 25% yield)

^{31}P-NMR (D_2O, internal standard: 85% H_3PO_4): δ -5.41, -10.31, -18.60

^{1}H-NMR (D_2O/MeOD = 2:1, Watergate solvent suppression): δ 8.17 (d, 1H, J = 7.6 Hz), 7.91 (d, 1H, J = 9.2 Hz), 7.7~7.5 (m, 2H), 7.1~7.5 (m, 9H), 6.07 (d, 1H, J = 9.2 Hz), 5.95 (t, 1H), 5.25 (s, 1H), 4.3~4.0 (m, 3H), 2.94 (t, 2H) , 2.47 (t, 2H), 2.16 (t, 2H)

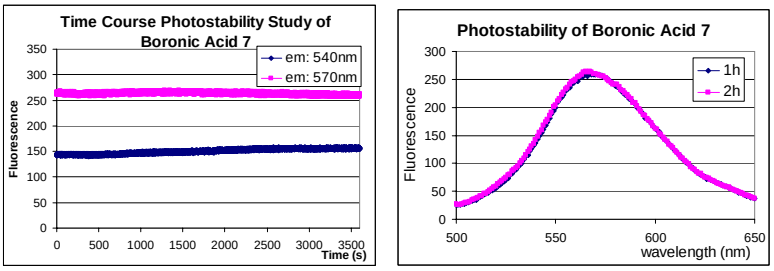
MS (ESI-): *m/z* 1032.3 (100) $[M-H-2H_2O-HPO_3]^-$, 1112.3 (20) $[M-H-2H_2O]^-$, 1134.3 $[M-H-3H_2O+MeOH]^-$, 1054.3 $[M-H-3H_2O+MeOH-HPO_3]^-$

MS (MALDI linear mode): *m/z* 1112.57 (100) $[M-H-2H_2O]^-$, 1032.97 (45) $[M-H-2H_2O-HPO_3]^-$

Exact mass: $[M-H-2H_2O]^-$ $C_{46}H_{42}BN_9O_{18}P_3$ calc. 1112.1954; found 1112.1940;

2. Photo stability test of boronic acid 7:

Solution of boronic acid **7** (1×10^{-6} M) in 0.1 M phosphate buffer at pH 7.4 was prepared. Using the Time Course function of Shimadzu RF-5301 PC spectrofluorometer, excitation wavelength was fixed at 490 nm. Both excitation and emission slits were set at 5 nm. The fluorescence intensity measurements were taken every 0.3 s at two wavelengths (540 and 570 nm) and the fluorescence was monitored for 3600 s [Fig. S1 (a)]. The entire fluorescence spectrum was scanned at the end point [Fig. S1 (b)]. It can be seen that the fluorescence intensity was constant at both tested emission wavelength (540 and 570 nm) and the fluorescence profiles overlap very well after constant irradiation for 1 h and 2 h.



(a) **(b)**

Fig. S1 Photostablity studies of boronic acid 7

3. NB-TTP Fluorescence binding tests:

Solutions of NB-TTP (1×10^{-6} M) and NB-TTP (1×10^{-6} M) with D-fructose (0.1 M) were prepared in 0.1 M phosphate buffer at pH 7.4, respectively. These two solutions were mixed in ratios to give the desired D-fructose concentrations (10^{-3} to 0.1 M). 3 ml of each mixed solution was used to test the fluorescence intensity several minutes after mixing. Eight points were collected for the calculation of the apparent binding constant K_a assuming a 1:1 complex formation mechanism. These studies were run in triplicate.

The binding constants were determined using the following equation:

$$I_0/(I-I_0)=(\varepsilon_B/\varepsilon_{B\text{-}S}K_a)*1/[\text{fructose}]+\varepsilon_B/\varepsilon_{B\text{-}S}$$

where I_0 is fluorescence intensity of NB-TTP solution; I is NB-TTP fluorescence intensity upon adding sugar, ε_B is fluorescence correlation factor of NB-TTP; ε_B-s is the fluorescence correlation factor of NB-TTP-fructose complex; K_a is the binding constant; [fructose] is the sugar concentration.

4. Primer extension using the Klenow fragment for MALDI-TOF-MS studies:

14-nt Primer (16 μM), 21-nt template (6.8 μM), Klenow (0.1 units/μl), TTP (0.3 mM) or NB-TTP (0.3 mM), and three other dNTPs (0.3 mM each) in total volume of 100 μl were incubated at 37°C for an hour. The prepared DNAs were purified using Microcon YM-3 centrifugal filter (Millipore Corp.) to remove dNTPs and other low molecular weight molecules. The harvested DNA was further purified by 5M NH_4OAc/ethanol precipitation (three times) as a way to replace metal ions with ammonium as the phosphate counterions.[3] The DNA pellet was dissolved in 10 μl for submission for MALDI-TOF-MS analysis. The oxidative deborylated NB-TTP-DNA sample was prepared by adding 0.25 μl of H_2O_2 solution (100 mM) into 5 μl of NB-TTP DNA solution. The MALDI mass spectral results with insulin as the internal standard are shown as following:

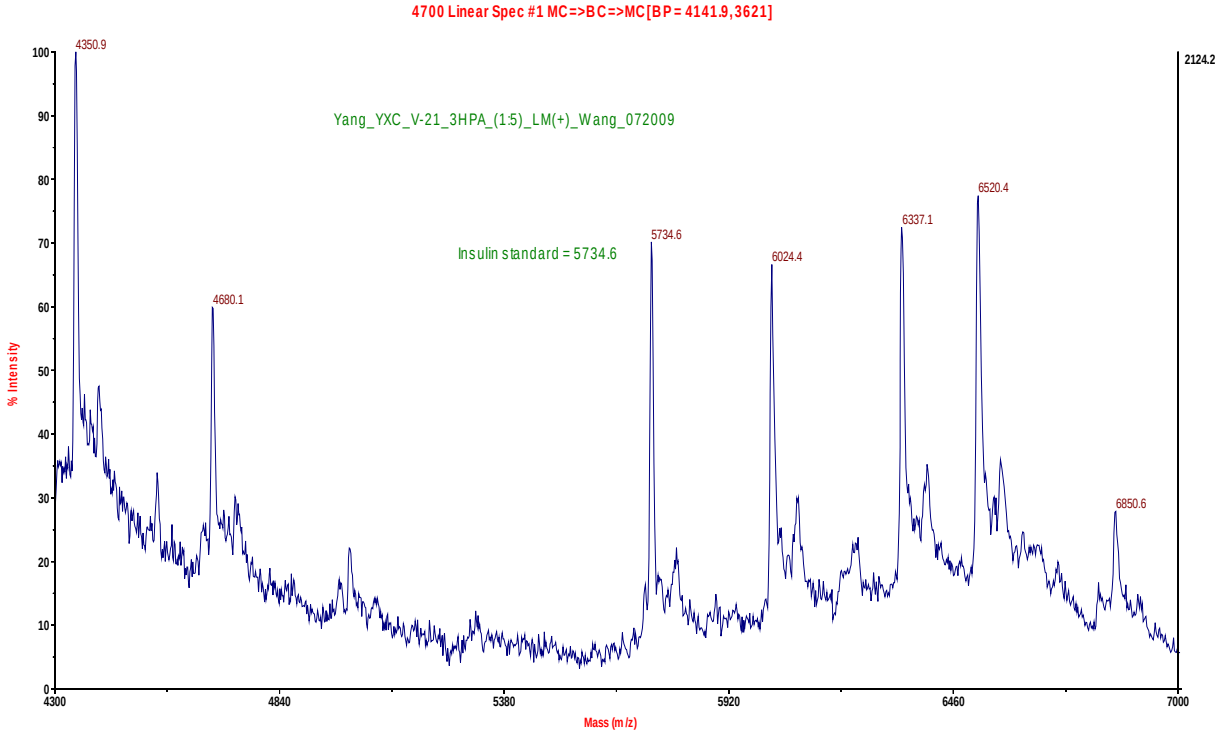


Fig. S2.1 MALDI-TOF-MS of primer extension using natural TTP

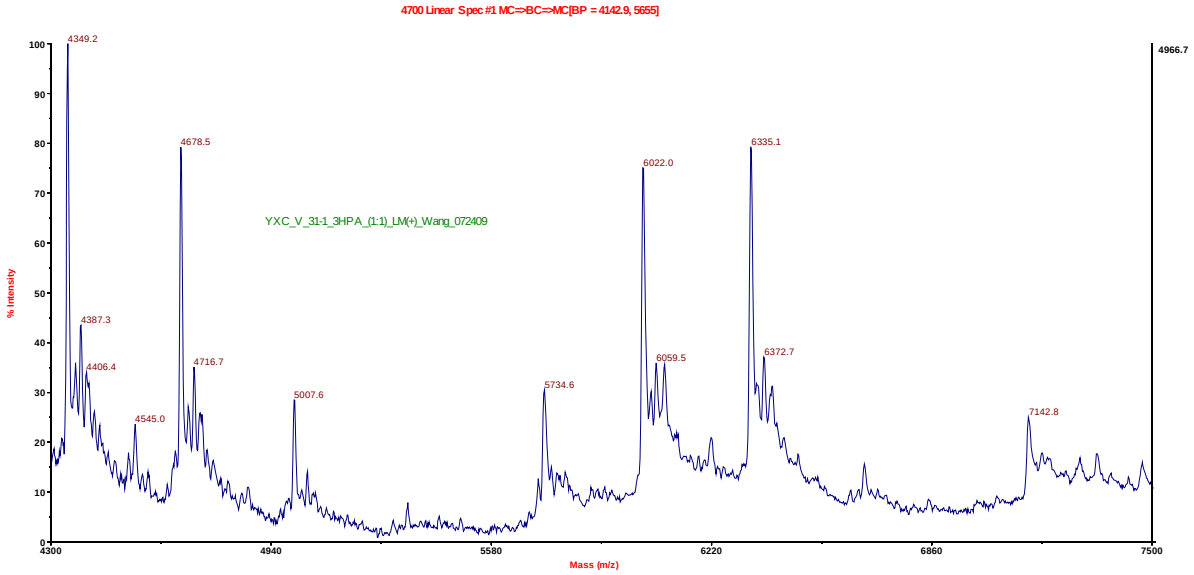


Fig. S2.2 MALDI-TOF-MS of primer extension using NB-TTP

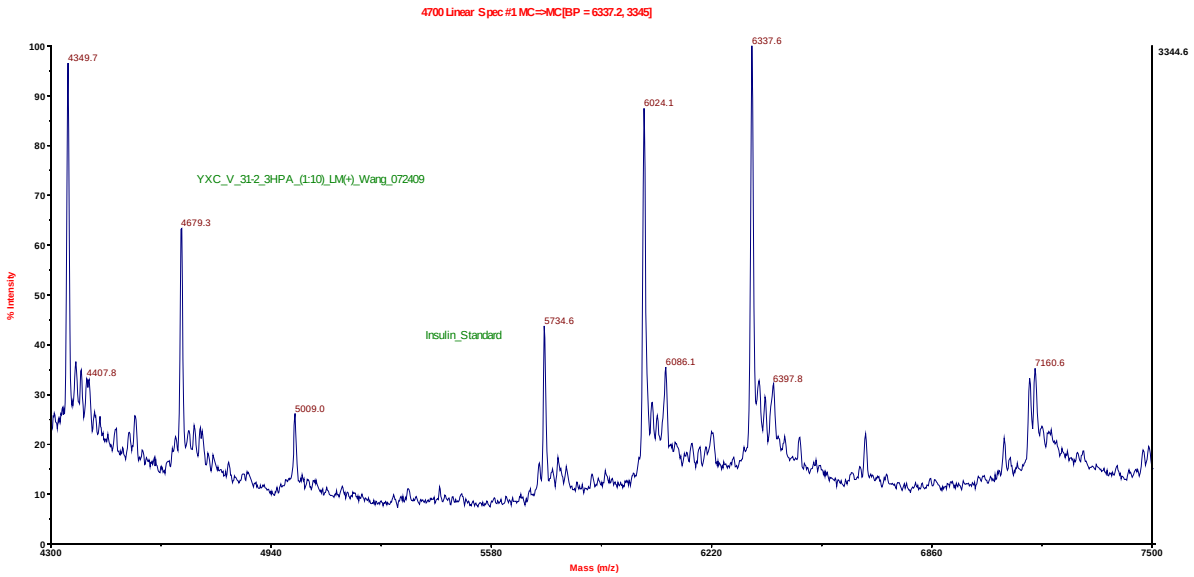


Fig. S2.3 MALDI-TOF-MS of primer extension using NB-TTP, followed by treatment with H_2O_2

5. PAGE analysis of primer extension:

A mixture of 14-nt primer DNA (50 μM), T4 polynucleotide kinase (0.5 units/μl, Biolabs, Inc.) and γ-^{32}P-ATP (0.6 μl, from Perkin-Elmer Corp.) in 12 μl T4 kinase buffer solution was incubated at 37 °C for 1 h. The ^{32}P-labeled DNA was purified using Microcon YM-3 centrifugal filter (Millipore Corp.) to remove low molecular weight molecules. Using a similar primer extension protocol as previously described, the ^{32}P-labeled primer alone, reaction mixture without enzyme, reaction mixture without dNTPs, reaction mixture with both enzyme and natural dNTPs and reaction mixture with NB-TTP, the other 3 dNTPs and enzyme were incubated at 37 °C for 1 h. The reactions were quenched with 2× DNA loading dye. 3 ul of samples from each reaction were taken and run on 15% PAGE at 300V for 3 h. Later, the gel was isolated, fixed and dried. The film was developed after autoradiography with the dried gel for overnight.

6. N**B-TTP-DNA Fluorescence binding test:**

1) Double stranded NB-TTP-DNA binding test:

Primer (7 μM), Template 0~7 (7 μM), Klenow (0.1 units/μl), NB-TTP (125 μM), and three other dNTPs (0.3 mM each) in total volume of 200 μl were incubated at 37 °C for an hour. After purification of the DNA through membrane filtration and NH_4OAc/ethanol precipitation three times, the orange-color DNA pellet was dissolved in 50 μl of H_2O. 45 μl of the NB-TTP-DNA solution was diluted by 4.5 ml 0.1 M phosphate buffer (pH 7.4) to make theoretical concentration of 0.5 μM solution. D-fructose was added into the DNA solution with concentration increasing from 10^{-3} to 0.1 M. 1 ml of solution was used to test the fluorescence intensity several minutes after mixing. Each test was triplicated (Fig. S3.1).

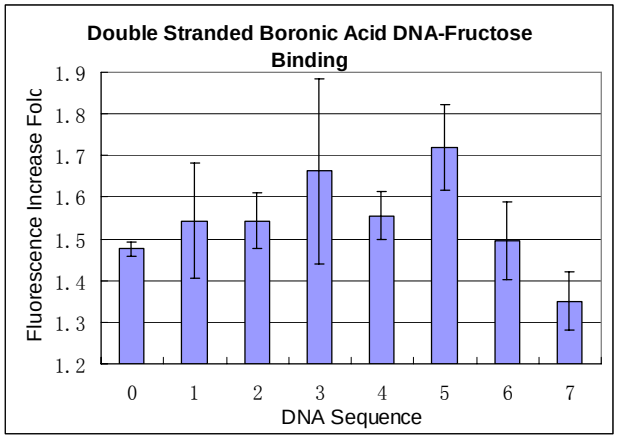


Fig. S3.1 Fluorescent binding tests of double stranded NB-TTP-DNA with D-fructose in 0.1M pH 7.4 phosphate buffer (λ_{ex}: 490 nm)

2) Single stranded NB-TTP-DNA binding test:

Primer (21 μM), Template 0~7 (7 μM), Klenow (0.1 units/μl), NB-TTP (125 μM), and three other dNTPs (0.3 mM each) in total volume of 200 μl were incubated at 37 °C for an hour. After purification of the DNA through membrane filtration and NH_4OAc/ethanol precipitation three times, the orange-color DNA pellet was dissolved in 50 μl of H_2O. 45 μl of the NB-TTP-DNA solution was heated at 90°C for 1 min, then quickly cooled down in ice bath and diluted by 2.5 ml 0.1 M phosphate buffer (pH 7.4) to make theoretical concentration of 0.5 μM solution. 0.5 ml of NB-TTP-DNA solution was mixed with 0.5 ml of phosphate buffer (blank) or 0.5 ml of D-fructose phosphate buffer solution (0.2 M), respectively. 1 ml of NB-TTP-DNA/fructose solution was tested the fluorescence intensity several minutes after mixing. The fluorescence intensity was divided by that of the blank to calculate the increasing fold. Each test was triplicated (Fig S3.2).

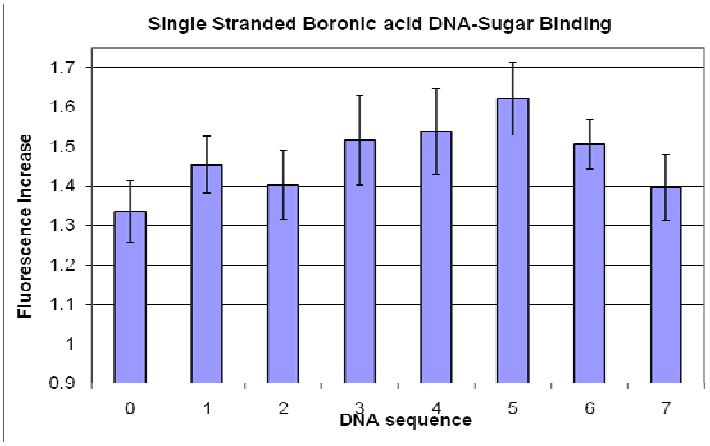


Fig. S3.2 Fluorescent binding tests of single stranded NB-TTP-DNA with saccharides in 0.1M pH 7.4 phosphate buffer (λ_{ex}: 490 nm)

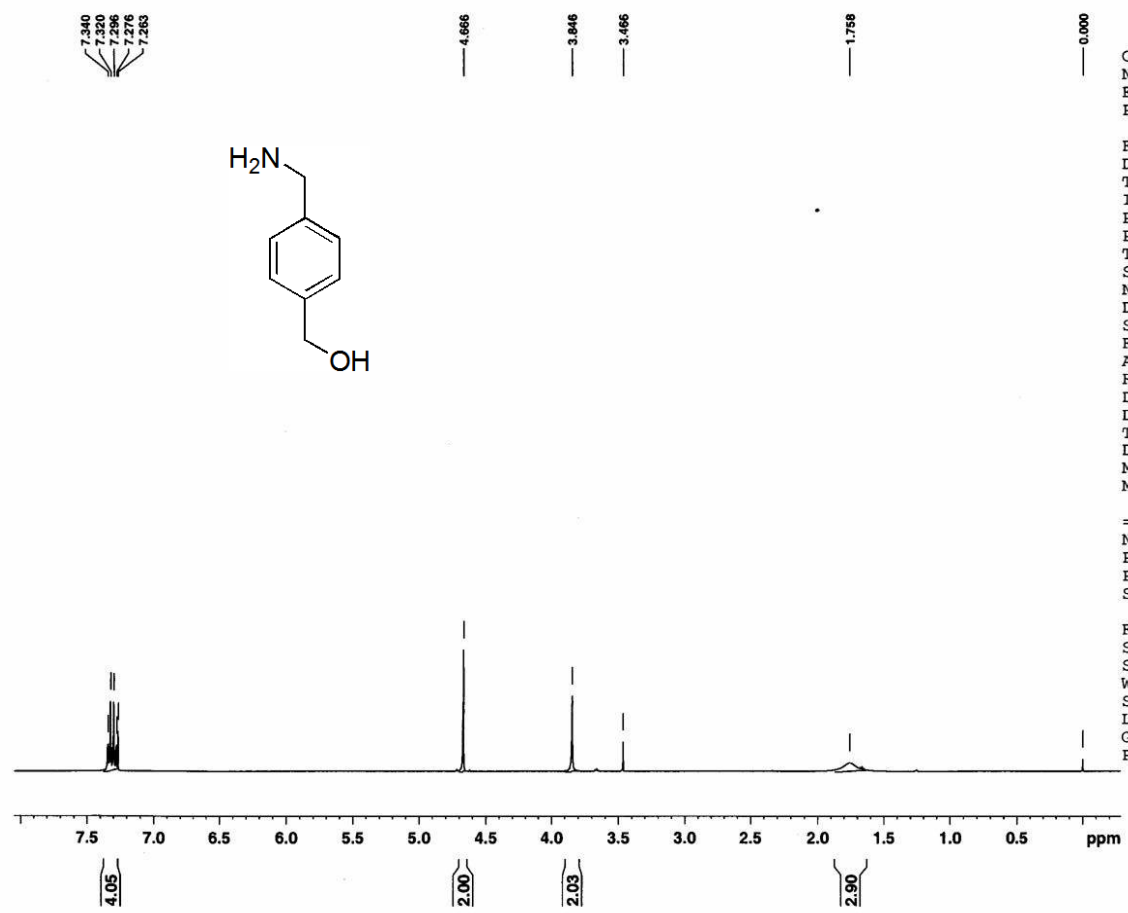
References:

(1) Wagner, A.; Breipohl, G.; Heitsch, H.; Gerhards, H.; Noelken, G.; Wirth, K.; Schoelkens, B.; Hoechst, A. G., Ed.; Ger. Offen.: German, 1997, p 28.

(2) Lee, J.; Kang, M.; Shin, M.; Kim, J. M.; Kang, S. U.; Lim, J. O.; Choi, H. K.; Suh, Y. G.; Park, H. G.; Oh, U.; Kim, H. D.; Park, Y. H.; Ha, H. J.; Kim, Y. H.; Toth, A.; Wang, Y.; Tran, R.; Pearce, L. V.; Lundberg, D. J.; Blumberg, P. M. *J. Med. Chem.* **2003**, *46*, 3116-3126.

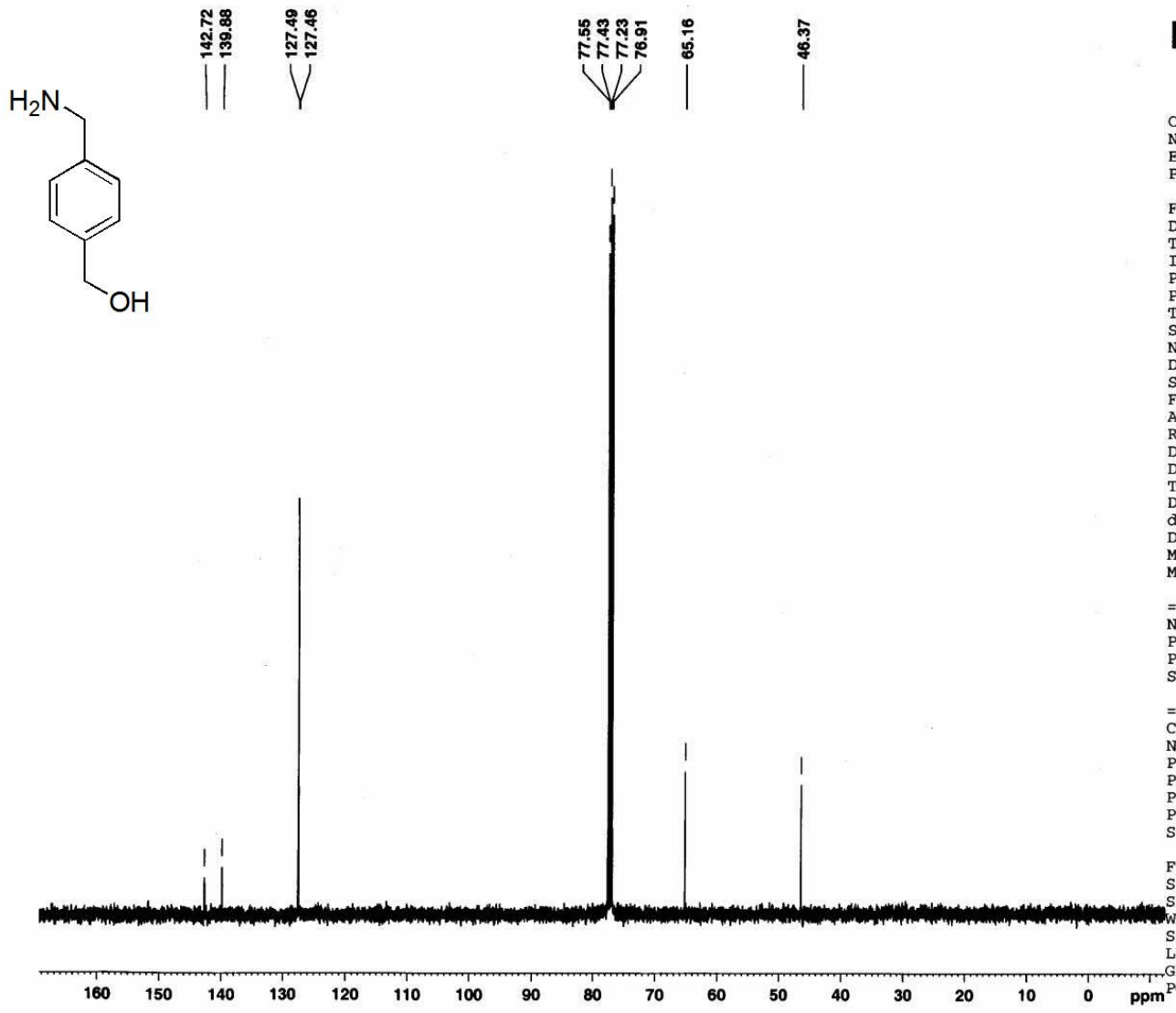
(3) Nordhoff, E.; Kirpekar, F.; Roepstorff, P. *Mass Spectrom. Rev.* **1996**, *15*, 67-138.

APPENDIX: NMR and MS Spectra

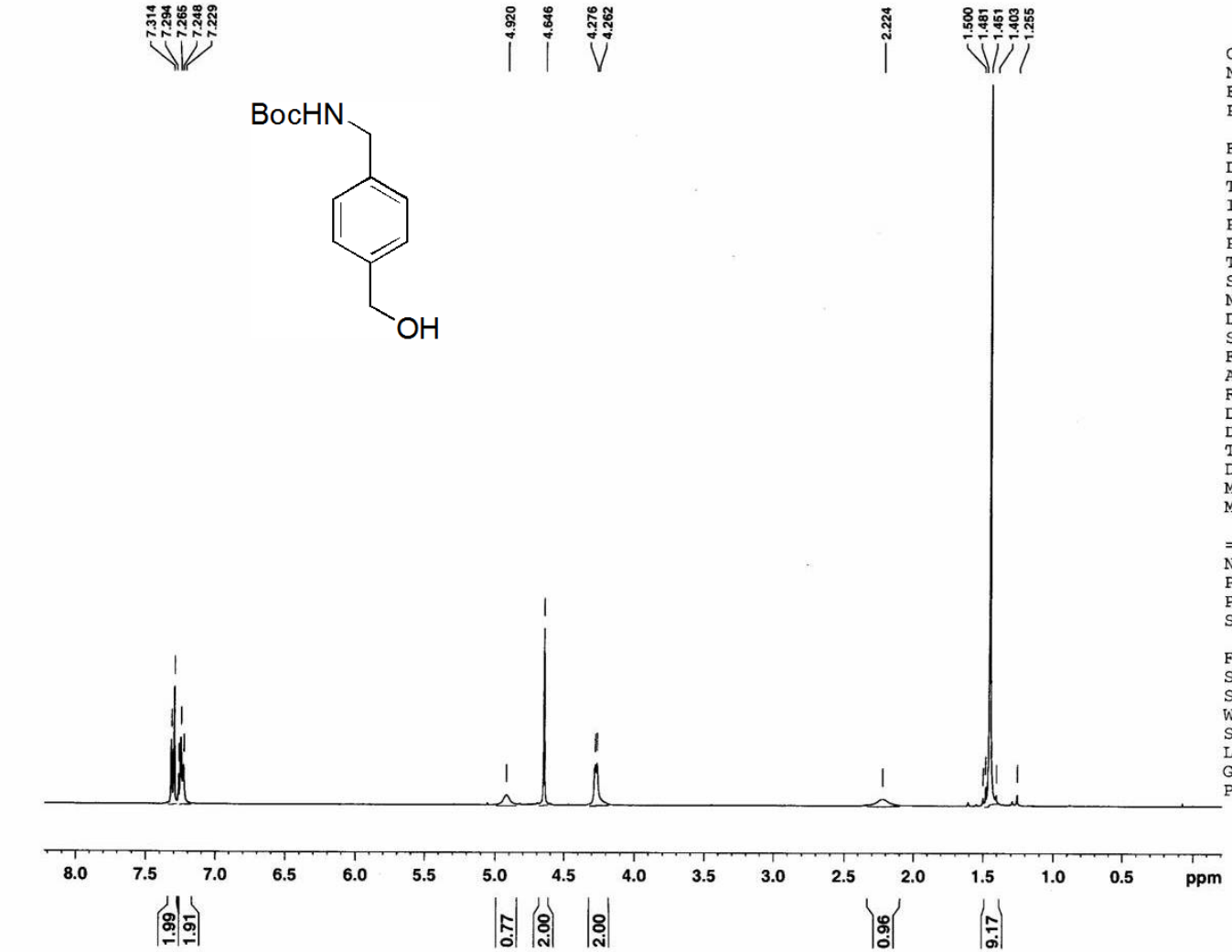


BRUKER

```
Current Data Parameters
NAME          YXC-III-68
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20080304
Time               11.03
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                 228.1
DW                60.400 usec
DE                 7.00 usec
TE                298.2 K
D1           1.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                12.80 usec
PL1                0.00 dB
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                 32768
SF          400.1300086 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                  1.00
```

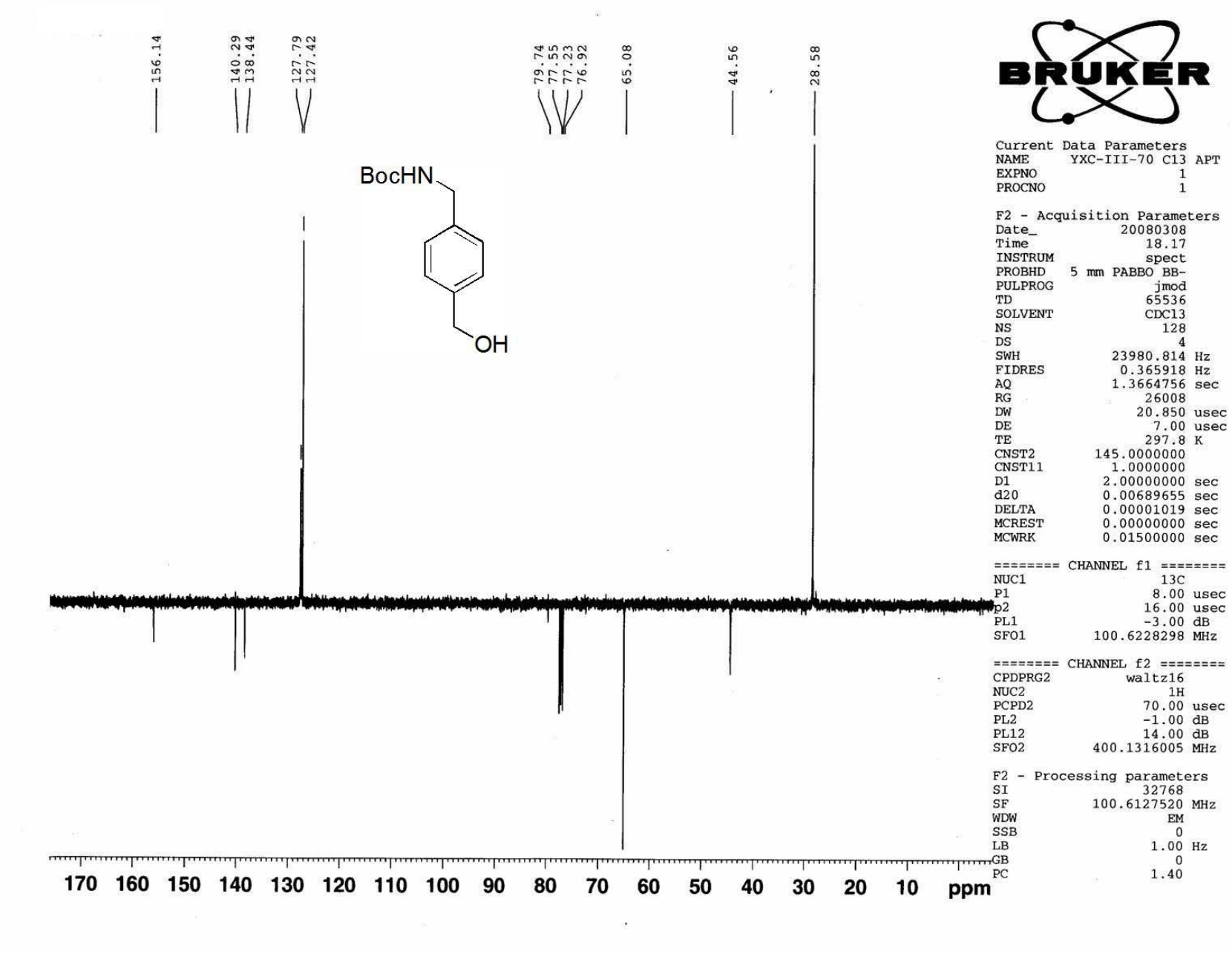


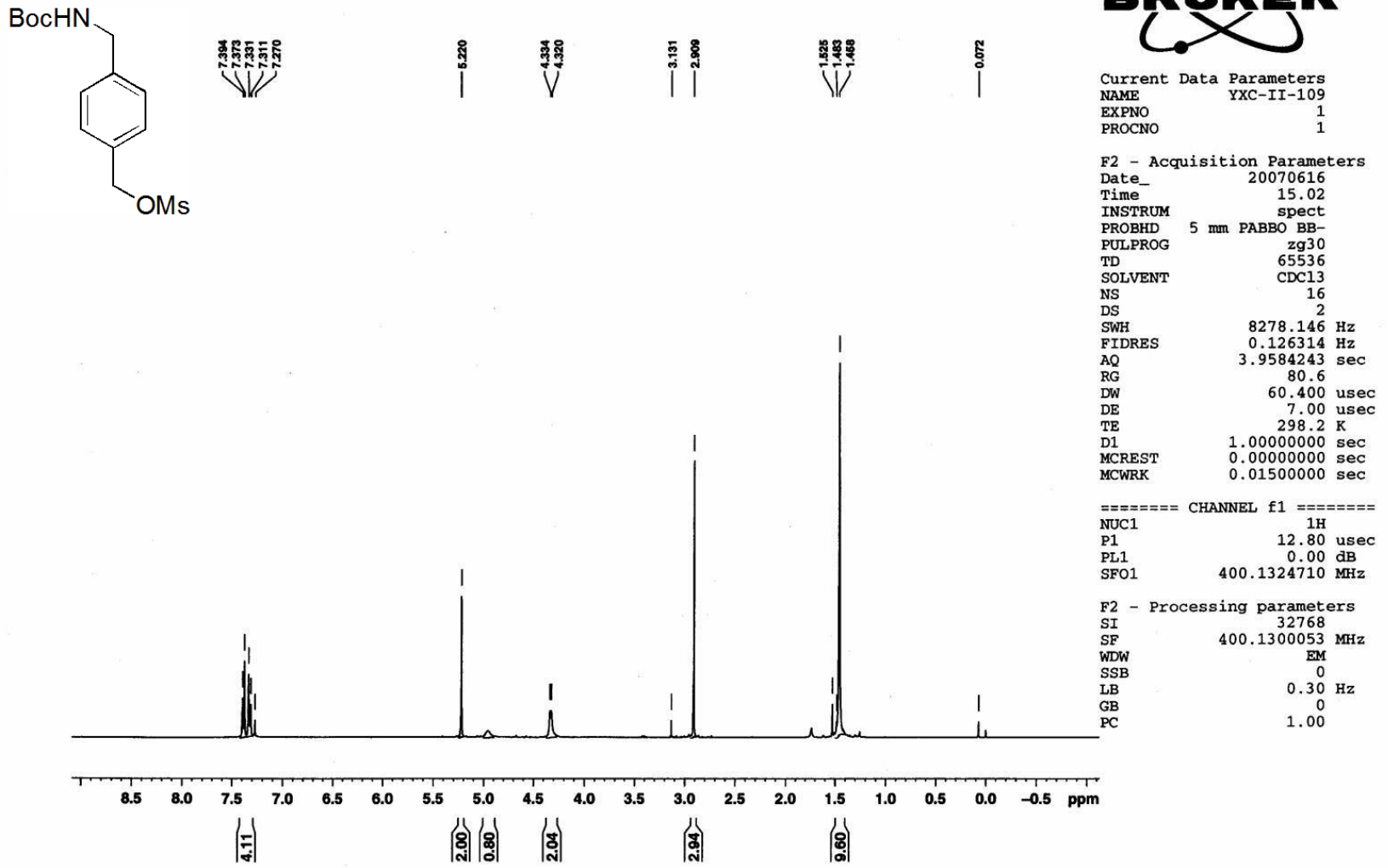
```
Current Data Parameters
NAME      YXC-III-68 C13
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20080304
Time              11.13
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG         zgpg30
TD                65536
SOLVENT             D2O
NS                  282
DS                    4
SWH           23980.814 Hz
FIDRES         0.365918 Hz
AQ            1.3664756 sec
RG                32768
DW               20.850 usec
DE                7.00 usec
TE               298.2 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  8.00 usec
PL1                -3.00 dB
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              70.00 usec
PL2                -1.00 dB
PL12               14.00 dB
PL13               14.00 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                  32768
SF           100.6127498 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```



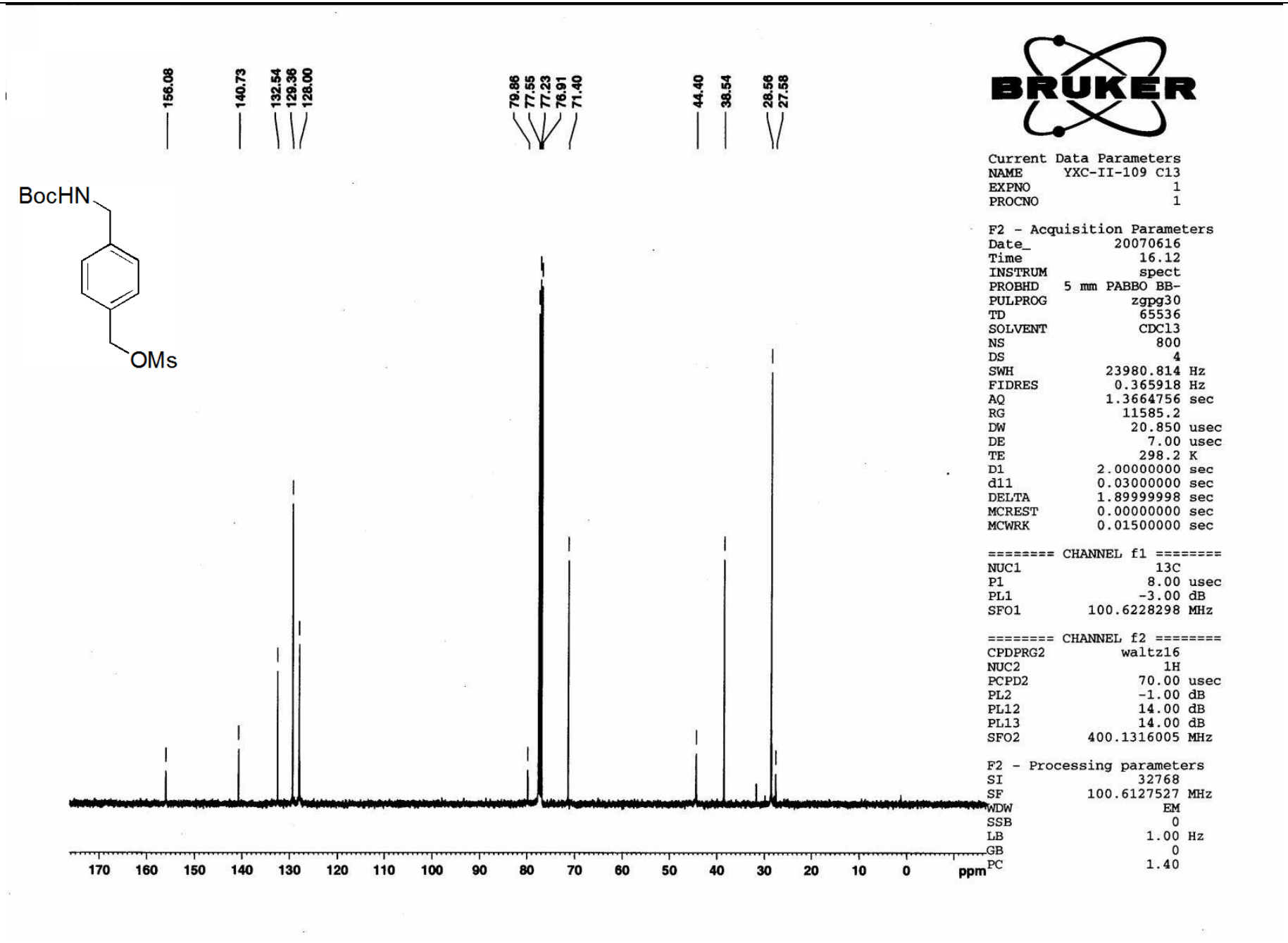
BRUKER

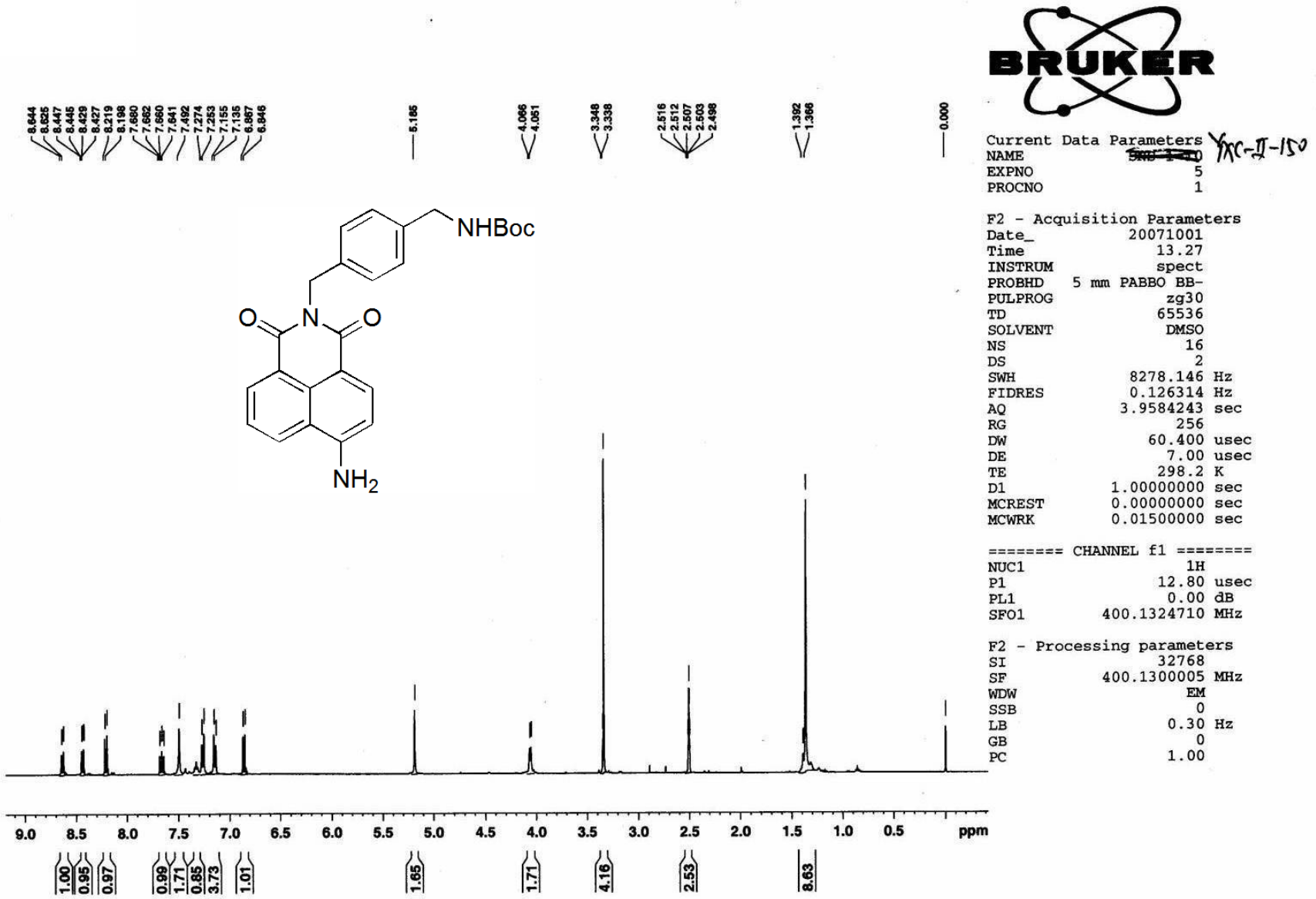
```
Current Data Parameters
NAME          YXC-III-70
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20080308
Time               18.08
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                  71.8
DW                60.400 usec
DE                 7.00 usec
TE                297.7 K
D1           1.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                 12.80 usec
PL1                 0.00 dB
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                 32768
SF          400.1300076 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```


156.14
140.29
138.44
127.79
127.42
79.74
77.55
77.23
76.92
65.08
44.56
28.58
BocHN
OH
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
BRUKER
Current Data Parameters
NAME YXC-III-70 C13 APT
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080308
Time 18.17
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG jmod
TD 65536
SOLVENT CDCl3
NS 128
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 26008
DW 20.850 usec
DE 7.00 usec
TE 297.8 K
CNST2 145.0000000
CNST11 1.0000000
D1 2.00000000 sec
d20 0.00689655 sec
DELTA 0.00001019 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 8.00 usec
p2 16.00 usec
PL1 -3.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 -1.00 dB
PL12 14.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127520 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40




```
Current Data Parameters
NAME                YXC-II-109
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20070616
Time                     15.02
INSTRUM                  spect
PROBHD    5 mm PABBO BB-
PULPROG                  zg30
TD                      65536
SOLVENT                 CDCl3
NS                         16
DS                          2
SWH                  8278.146 Hz
FIDRES               0.126314 Hz
AQ                   3.9584243 sec
RG                        80.6
DW                      60.400 usec
DE                       7.00 usec
TE                      298.2 K
D1                1.00000000 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                        1H
P1                       12.80 usec
PL1                       0.00 dB
SFO1              400.1324710 MHz

F2 - Processing parameters
SI                       32768
SF                400.1300053 MHz
WDW                         EM
SSB                          0
LB                        0.30 Hz
GB                           0
PC                        1.00
```


156.08
140.73
132.54
129.36
128.00
79.86
77.55
77.23
76.91
71.40
44.40
38.54
28.56
27.58
BocHN
OMs
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm
BRUKER
Current Data Parameters
NAME YXC-II-109 C13
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070616
Time 16.12
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 800
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 11585.2
DW 20.850 usec
DE 7.00 usec
TE 298.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 8.00 usec
PL1 -3.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 -1.00 dB
PL12 14.00 dB
PL13 14.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127527 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



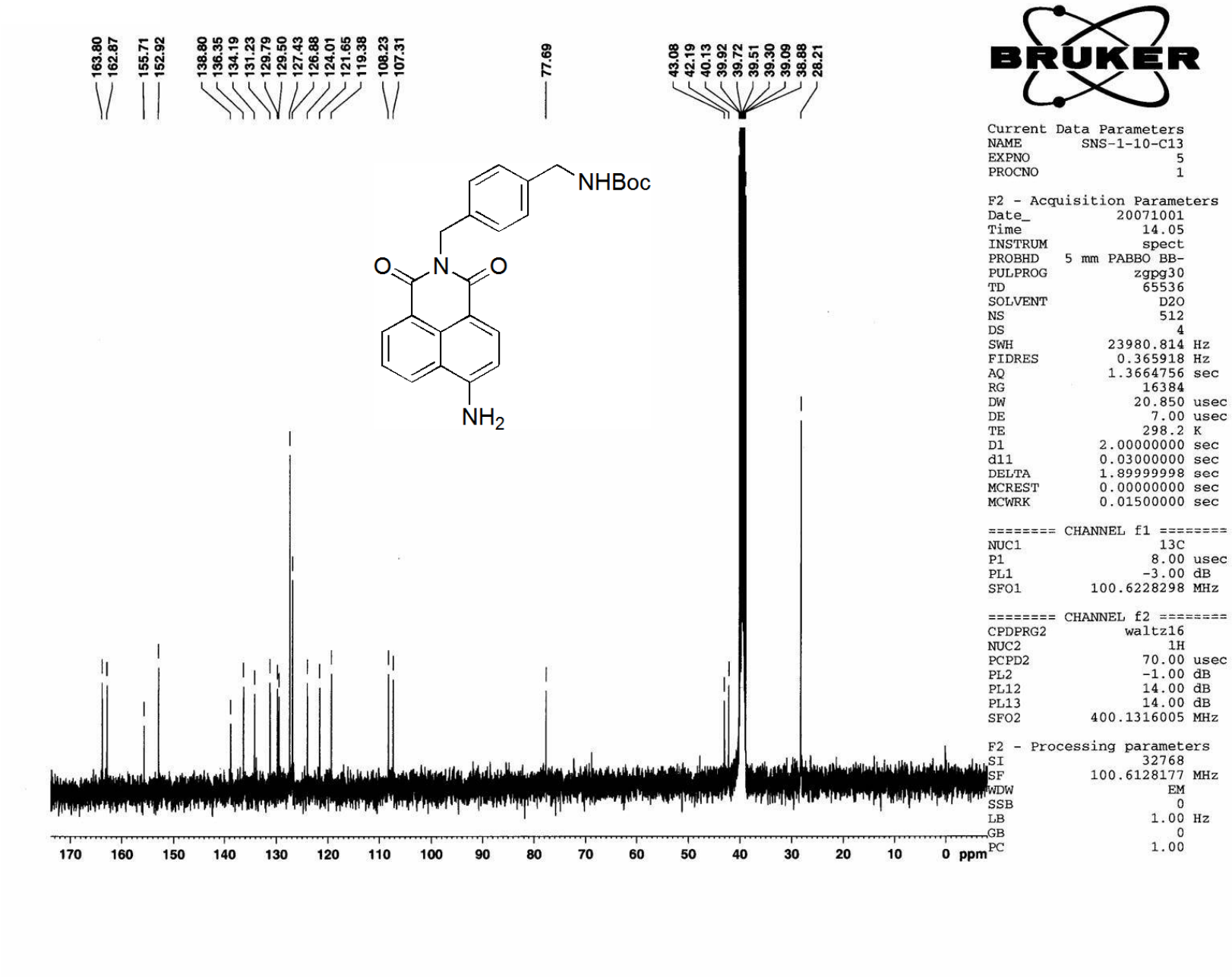
BRUKER

Current Data Parameters YXC-II-150
NAME ~~SNB-1750~~
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20071001
Time 13.27
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 256
DW 60.400 usec
DE 7.00 usec
TE 298.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

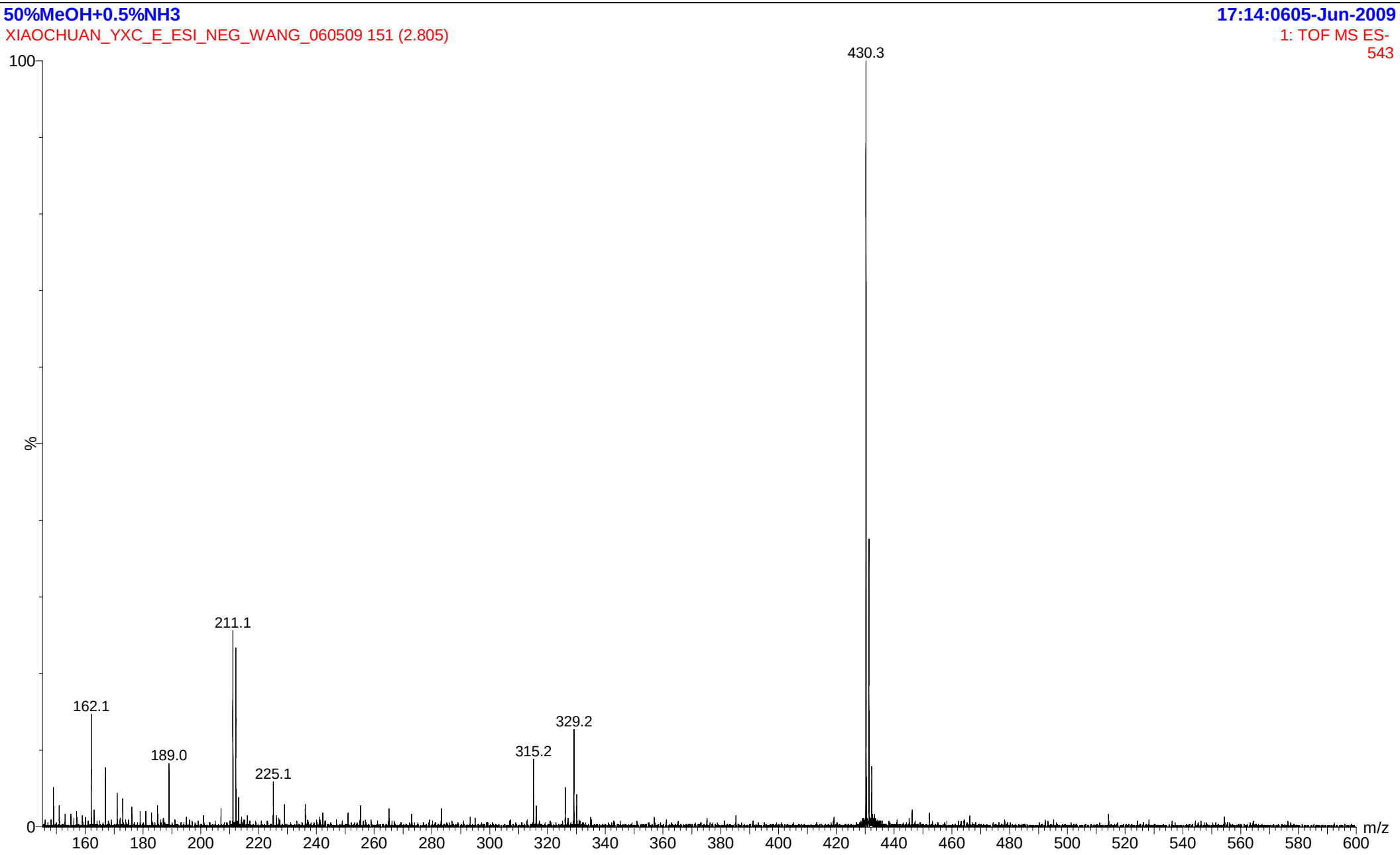
======== CHANNEL f1 ========
NUC1 1H
P1 12.80 usec
PL1 0.00 dB
SFO1 400.1324710 MHz

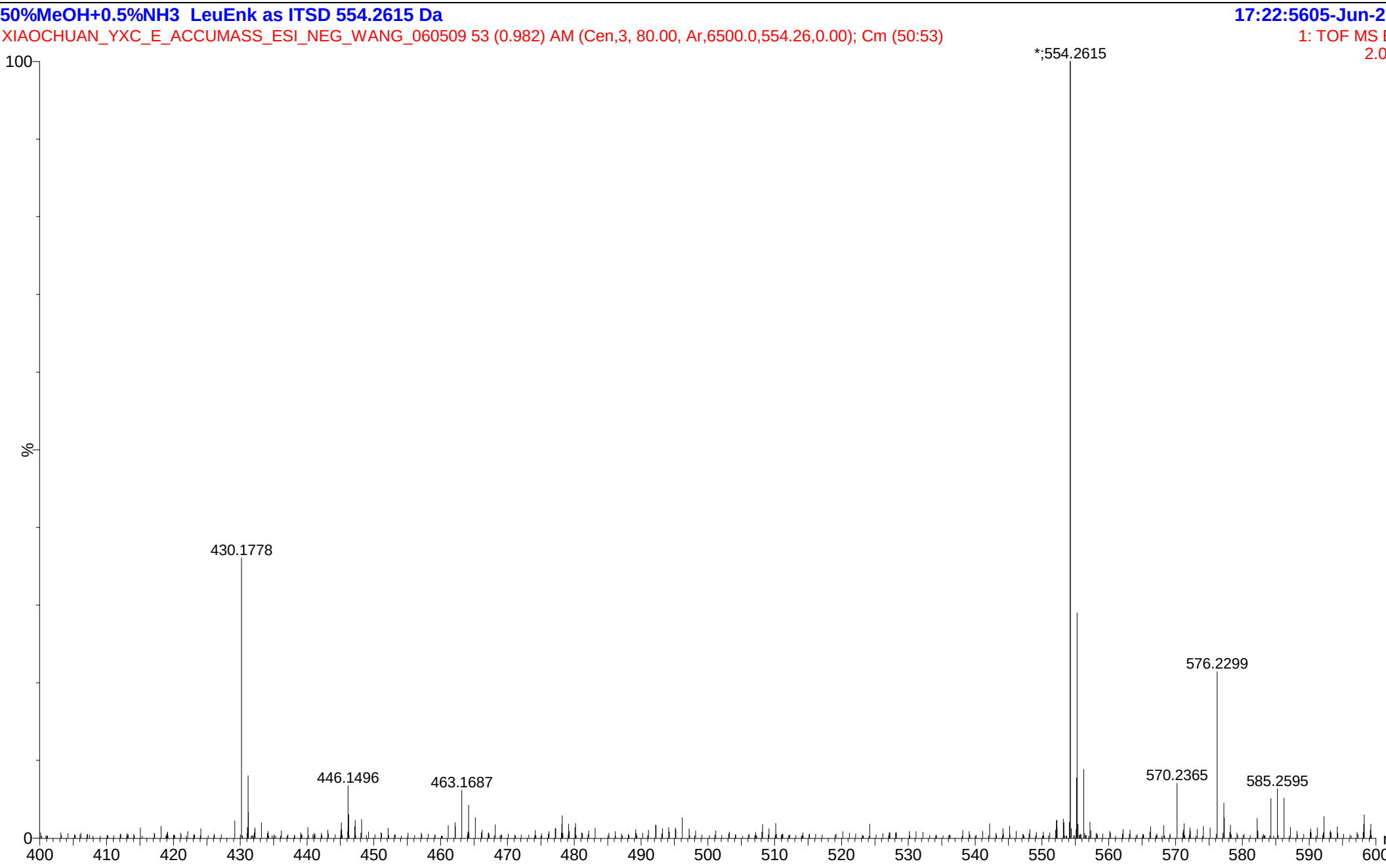
F2 - Processing parameters
SI 32768
SF 400.1300005 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

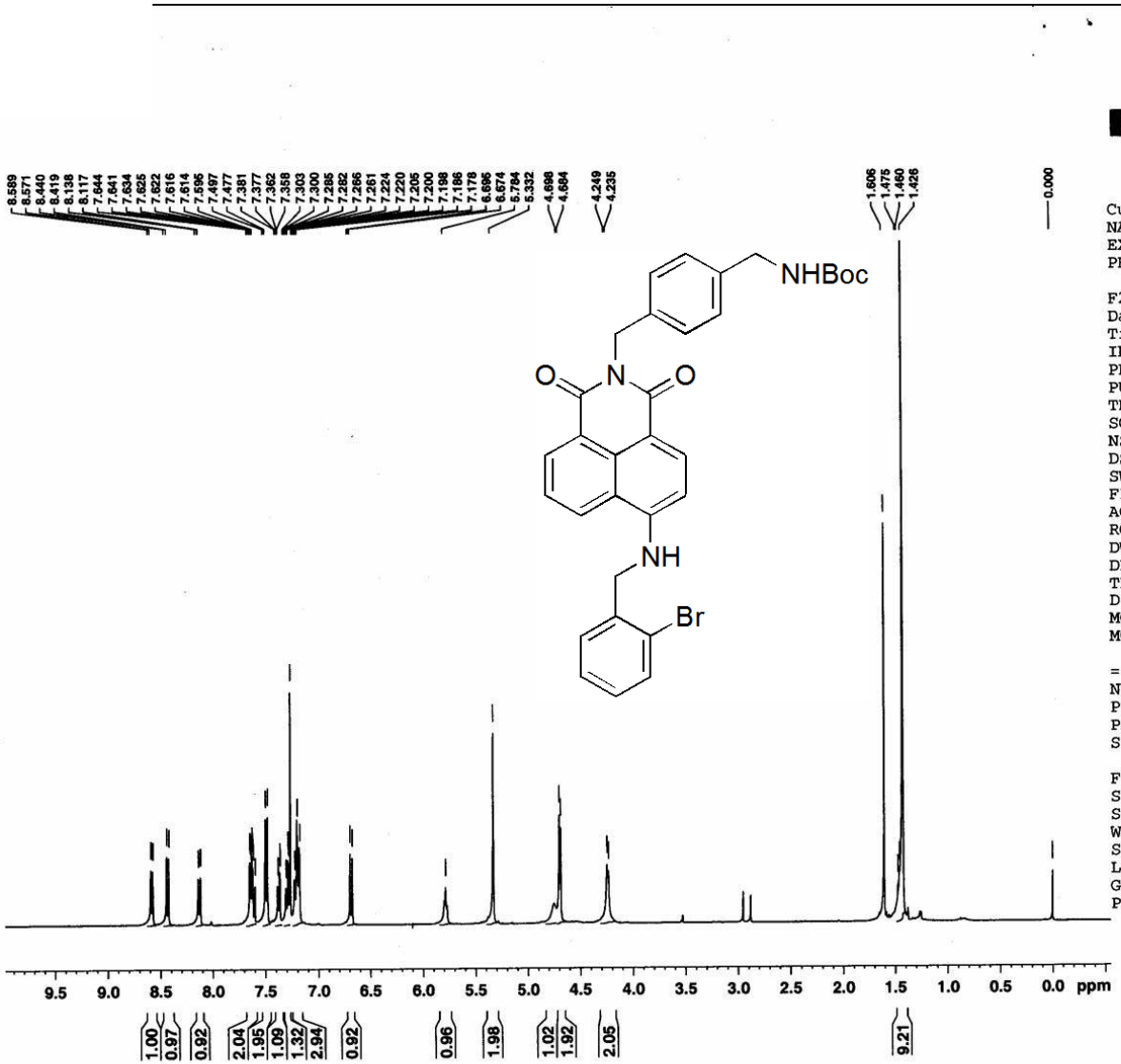


BRUKER

```
Current Data Parameters
NAME          SNS-1-10-C13
EXPNO                    5
PROCNO                   1

F2 - Acquisition Parameters
Date_             20071001
Time                 14.05
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT                D2O
NS                       512
DS                         4
SWH              23980.814 Hz
FIDRES           0.365918 Hz
AQ               1.3664756 sec
RG                   16384
DW                  20.850 usec
DE                   7.00 usec
TE                   298.2 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                   13C
P1                    8.00 usec
PL1                  -3.00 dB
SFO1          100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2                70.00 usec
PL2                  -1.00 dB
PL12                 14.00 dB
PL13                 14.00 dB
SFO2          400.1316005 MHz

F2 - Processing parameters
SI                    32768
SF            100.6128177 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.00
```


50%MeOH+0.5%NH3
17:14:0605-Jun-2009
XIAOCHUAN_YXC_E_ESI_NEG_WANG_060509 151 (2.805)
1: TOF MS ES-
543
100
%
0
430.3
211.1
162.1
329.2
315.2
189.0
225.1
m/z
160
180
200
220
240
260
280
300
320
340
360
380
400
420
440
460
480
500
520
540
560
580
600


50%MeOH+0.5%NH3 LeuEnk as ITSD 554.2615 Da
17:22:5605-Jun-2
XIAOCHUAN_YXC_E_ACCUMASS_ESI_NEG_WANG_060509 53 (0.982) AM (Cen,3, 80.00, Ar,6500.0,554.26,0.00); Cm (50:53)
1: TOF MS E
2.0
*;554.2615
430.1778
446.1496
463.1687
570.2365
576.2299
585.2595
100
%
0
400
410
420
430
440
450
460
470
480
490
500
510
520
530
540
550
560
570
580
590
600



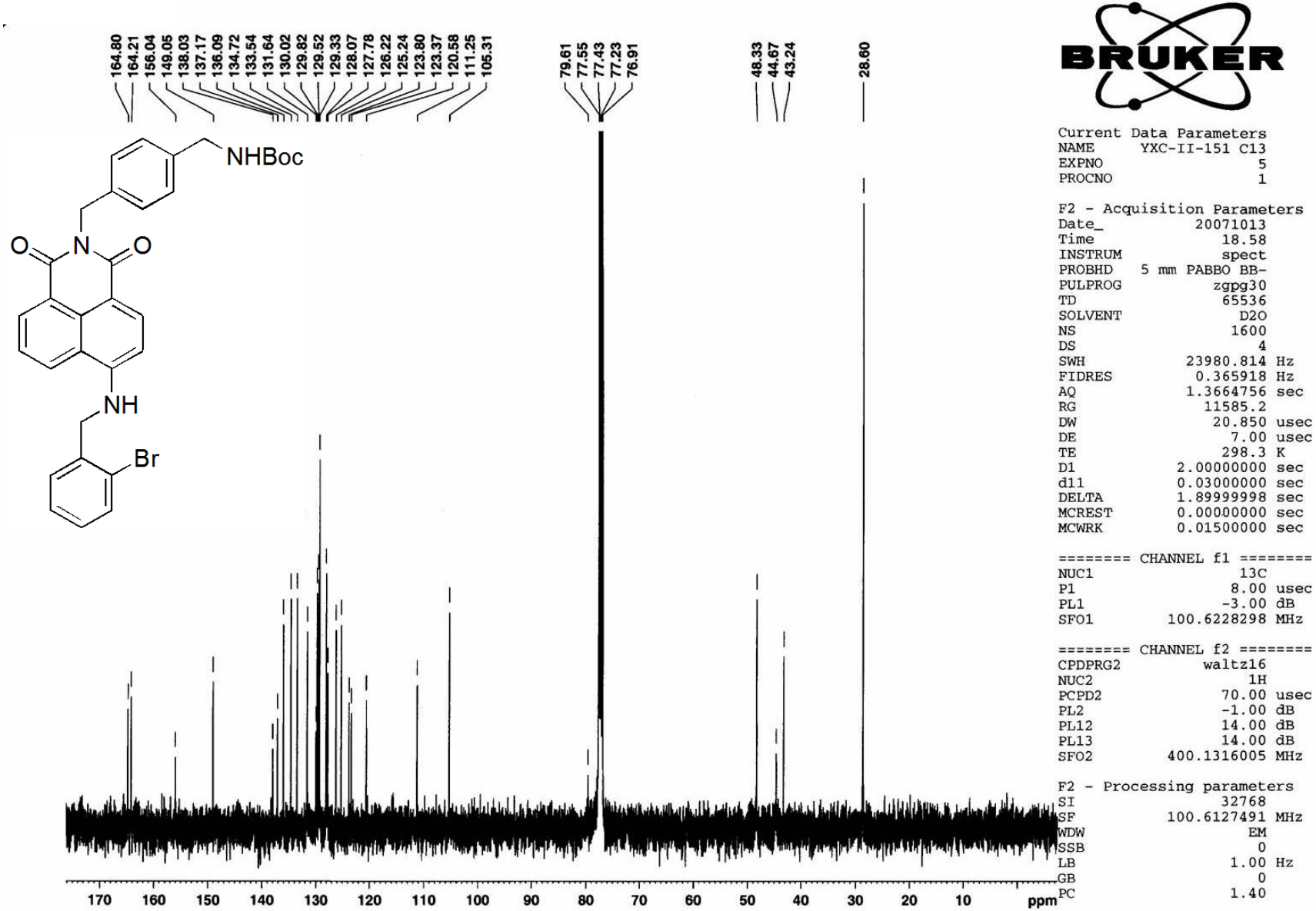
BRUKER

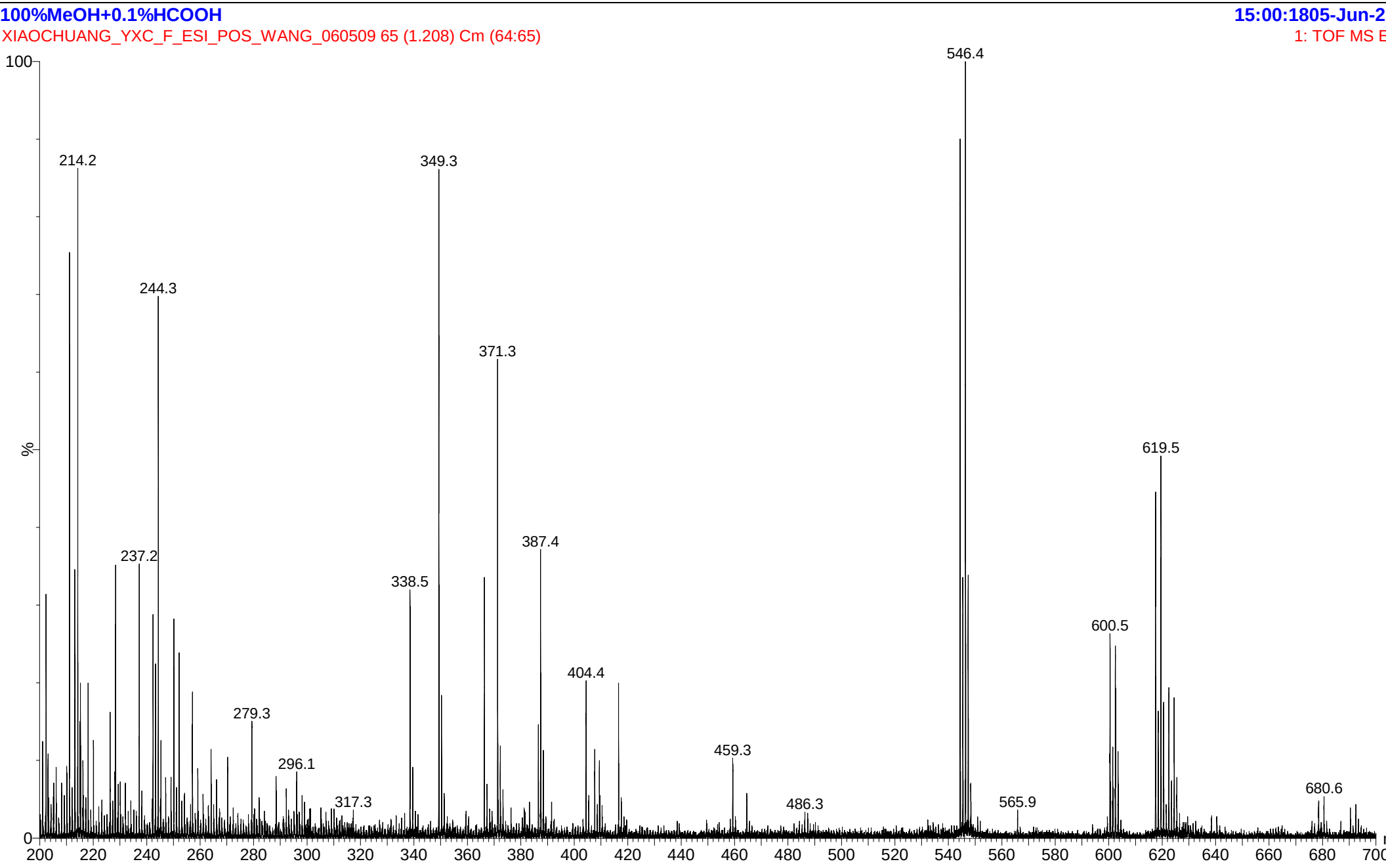
Current Data Parameters
NAME YXC-II-151
EXPNO 5
PROCNO 1

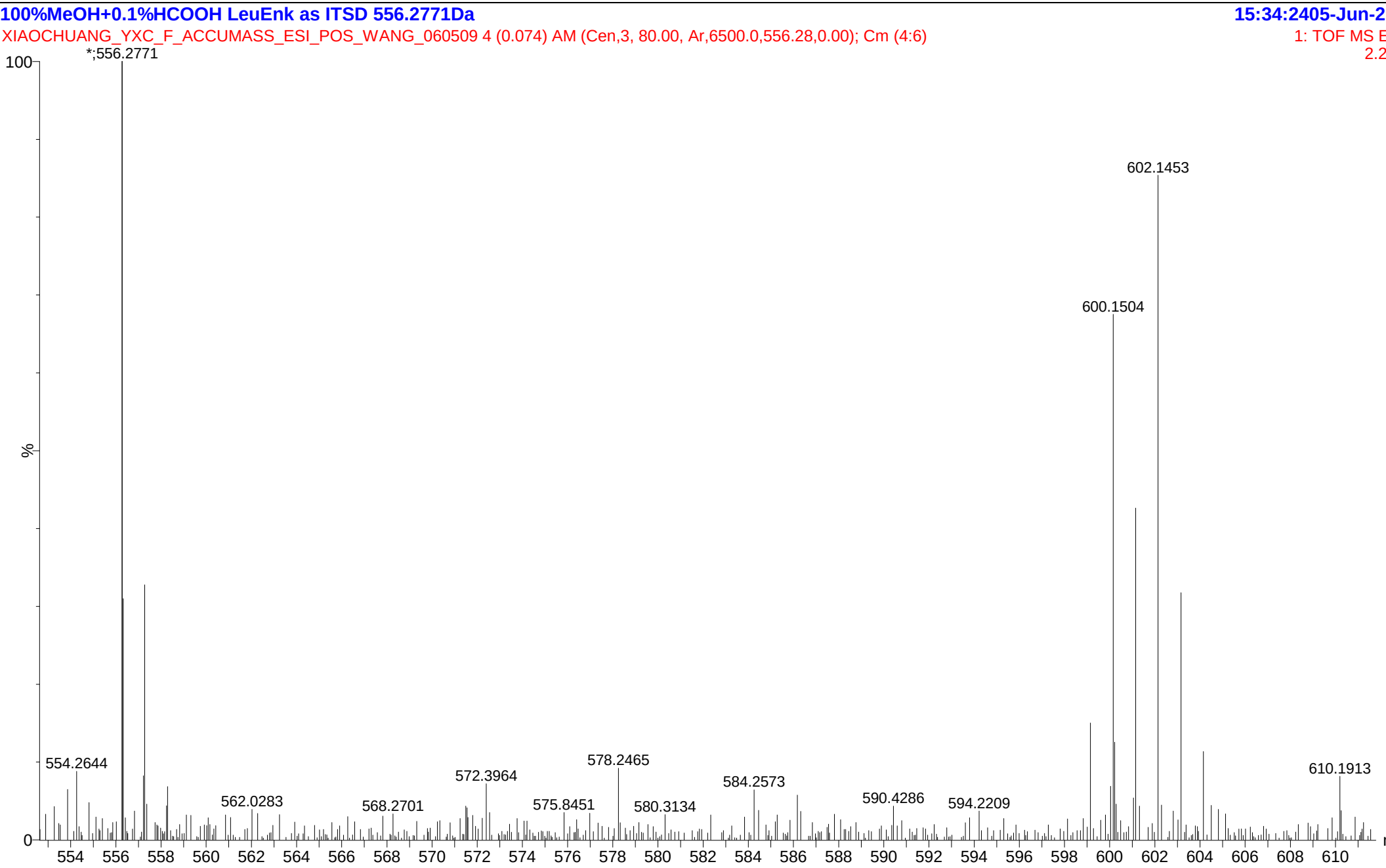
F2 - Acquisition Parameters
Date_ 20071013
Time 16.59
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 287.4
DW 60.400 usec
DE 7.00 usec
TE 298.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

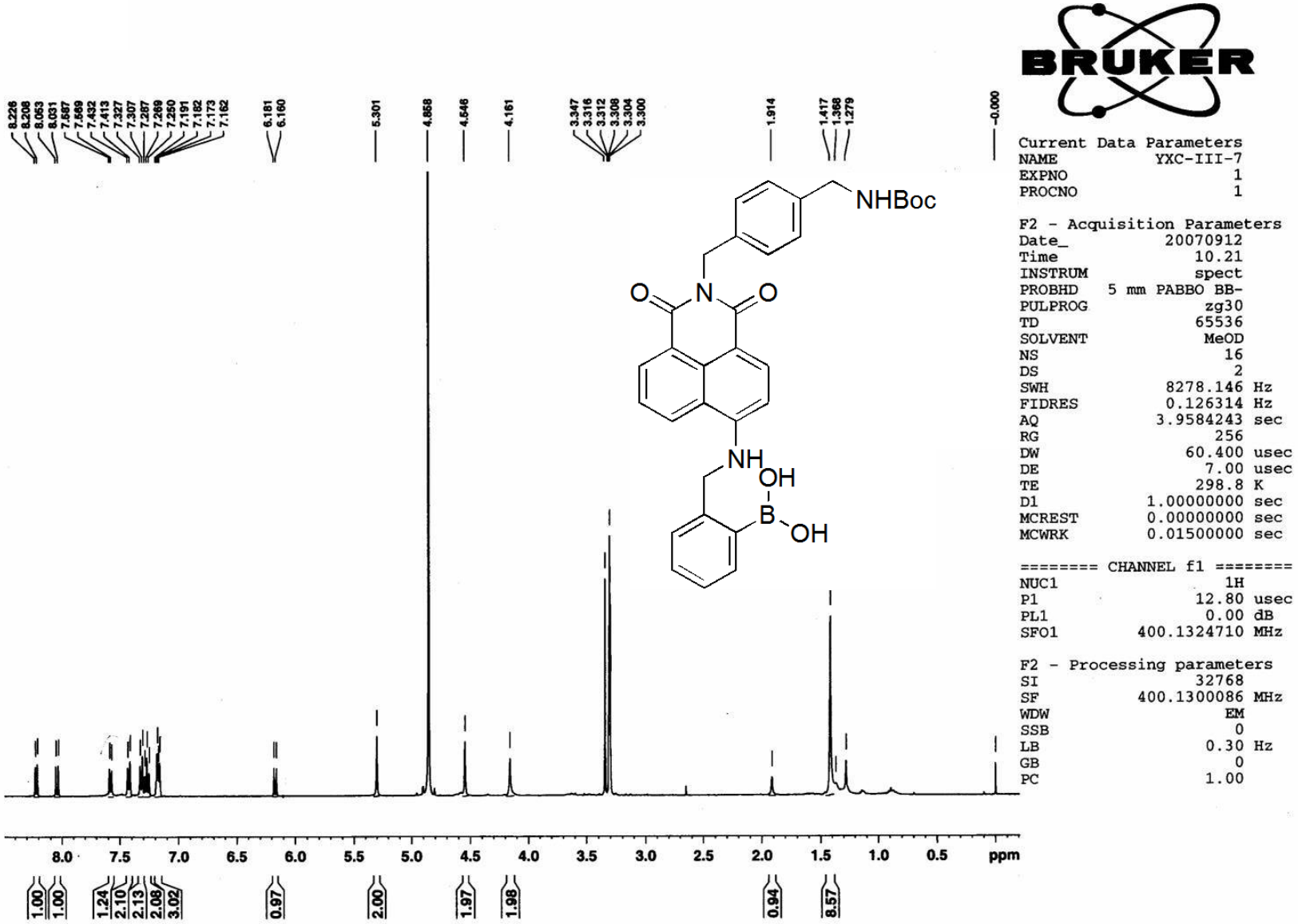
======== CHANNEL f1 ========
NUC1 1H
P1 12.80 usec
PL1 0.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300091 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


164.80
164.21
156.04
149.05
138.03
137.17
136.09
134.72
133.54
131.64
130.02
129.82
129.52
129.33
128.07
127.78
126.22
125.24
123.80
123.37
120.58
111.25
105.31
79.61
77.55
77.43
77.23
76.91
48.33
44.67
43.24
28.60
NHBoc
O
N
O
NH
Br
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm
BRUKER
Current Data Parameters
NAME YXC-II-151 C13
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20071013
Time 18.58
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT D2O
NS 1600
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 11585.2
DW 20.850 usec
DE 7.00 usec
TE 298.3 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 8.00 usec
PL1 -3.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 -1.00 dB
PL12 14.00 dB
PL13 14.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127491 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


100%MeOH+0.1%HCOOH
15:00:1805-Jun-2
XIAOCHUANG_YXC_F_ESI_POS_WANG_060509 65 (1.208) Cm (64:65)
1: TOF MS E
100
%
0
546.4
214.2
349.3
244.3
371.3
619.5
387.4
237.2
338.5
600.5
404.4
279.3
459.3
296.1
680.6
317.3
486.3
565.9
200
220
240
260
280
300
320
340
360
380
400
420
440
460
480
500
520
540
560
580
600
620
640
660
680
700


100%MeOH+0.1%HCOOH LeuEnk as ITSD 556.2771Da
15:34:2405-Jun-2
XIAOCHUANG_YXC_F_ACCUMASS_ESI_POS_WANG_060509 4 (0.074) AM (Cen,3, 80.00, Ar,6500.0,556.28,0.00); Cm (4:6)
1: TOF MS E
2.2
*;556.2771
602.1453
600.1504
554.2644
562.0283
568.2701
572.3964
575.8451
578.2465
580.3134
584.2573
590.4286
594.2209
610.1913
100
0
%
554 556 558 560 562 564 566 568 570 572 574 576 578 580 582 584 586 588 590 592 594 596 598 600 602 604 606 608 610



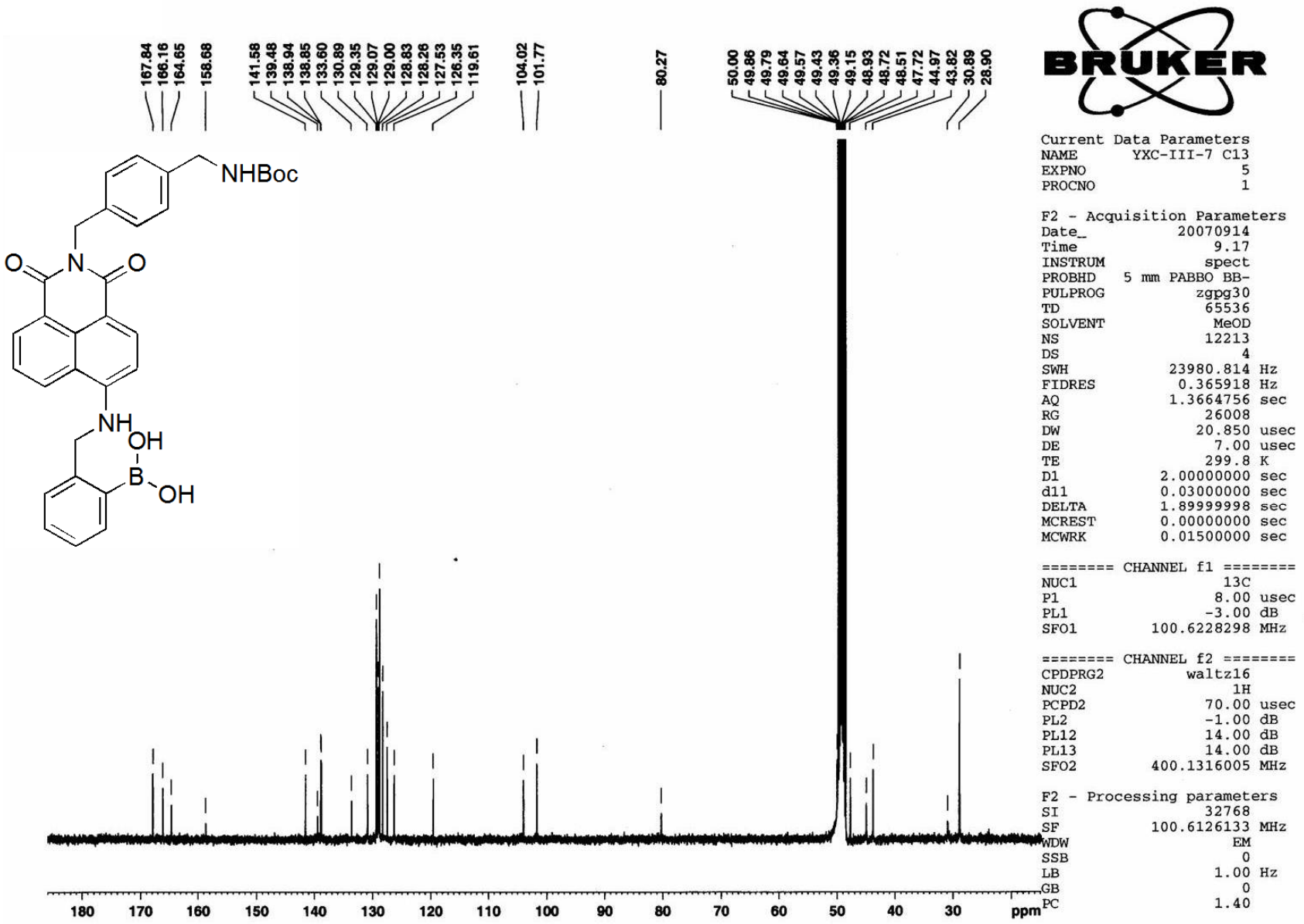
BRUKER

Current Data Parameters
NAME YXC-III-7
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070912
Time 10.21
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 256
DW 60.400 usec
DE 7.00 usec
TE 298.8 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 12.80 usec
PL1 0.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300086 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



BRUKER

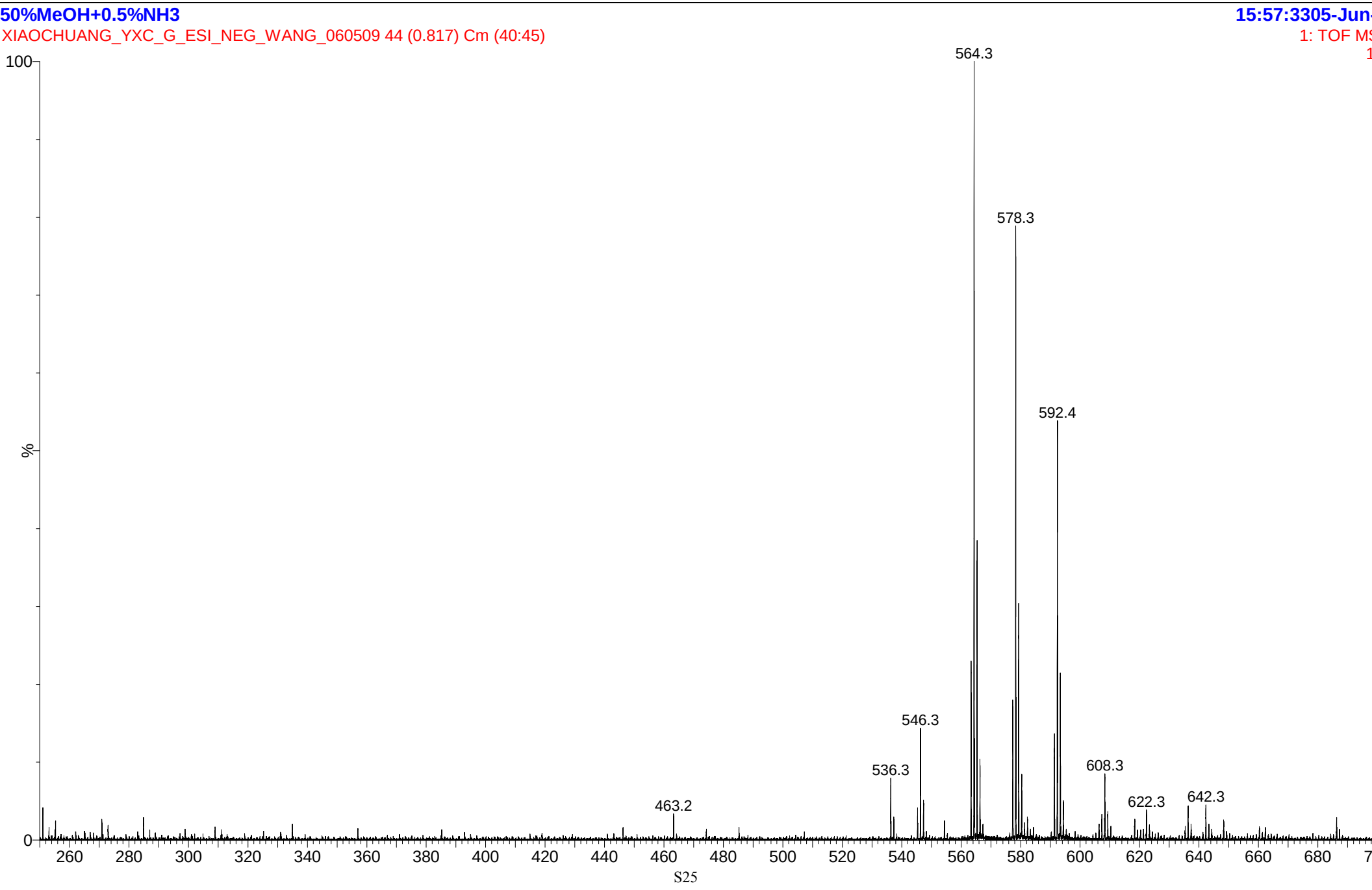
Current Data Parameters
NAME YXC-III-7 C13
EXPNO 5
PROCNO 1

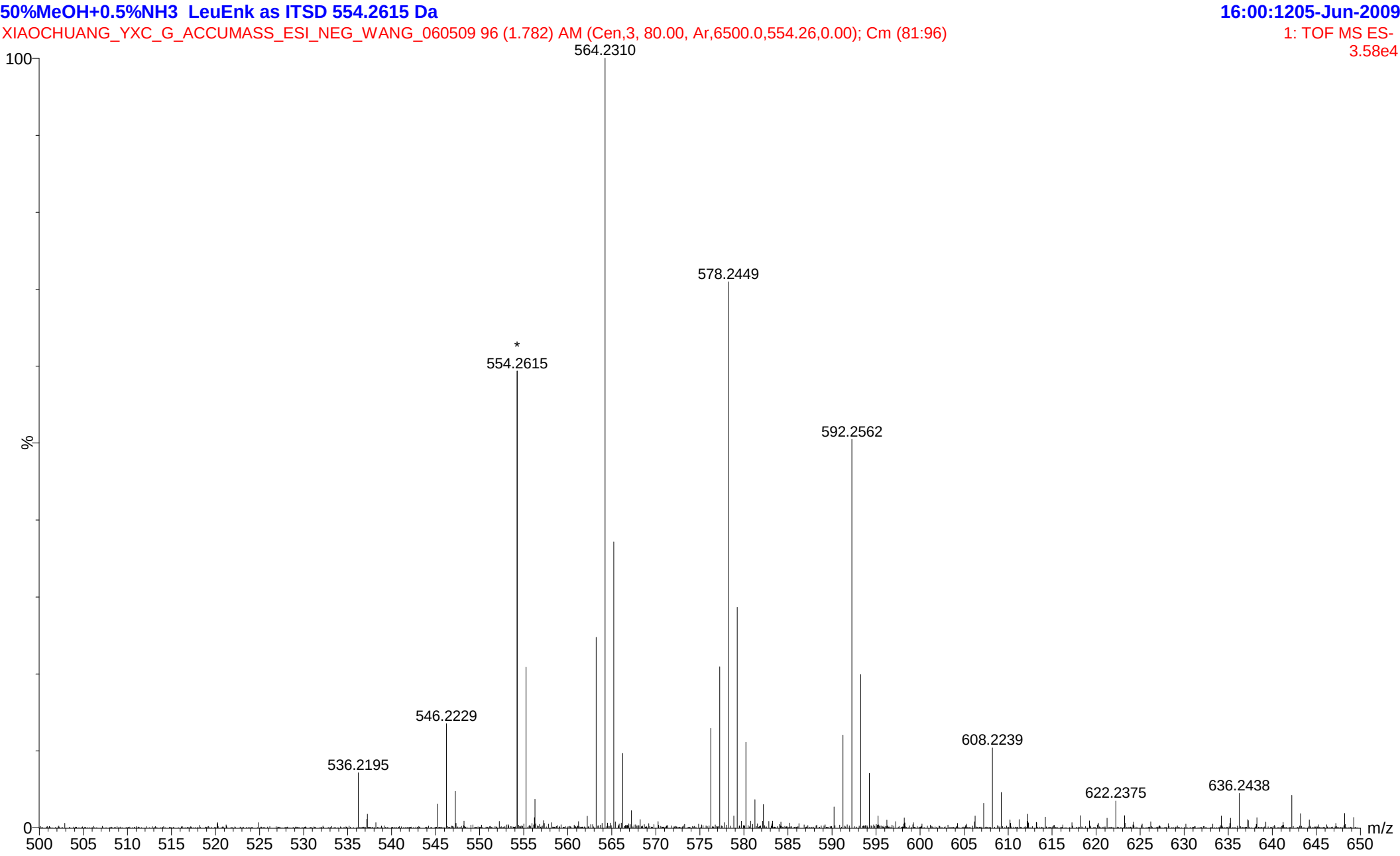
F2 - Acquisition Parameters
Date_ 20070914
Time 9.17
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 12213
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 26008
DW 20.850 usec
DE 7.00 usec
TE 299.8 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

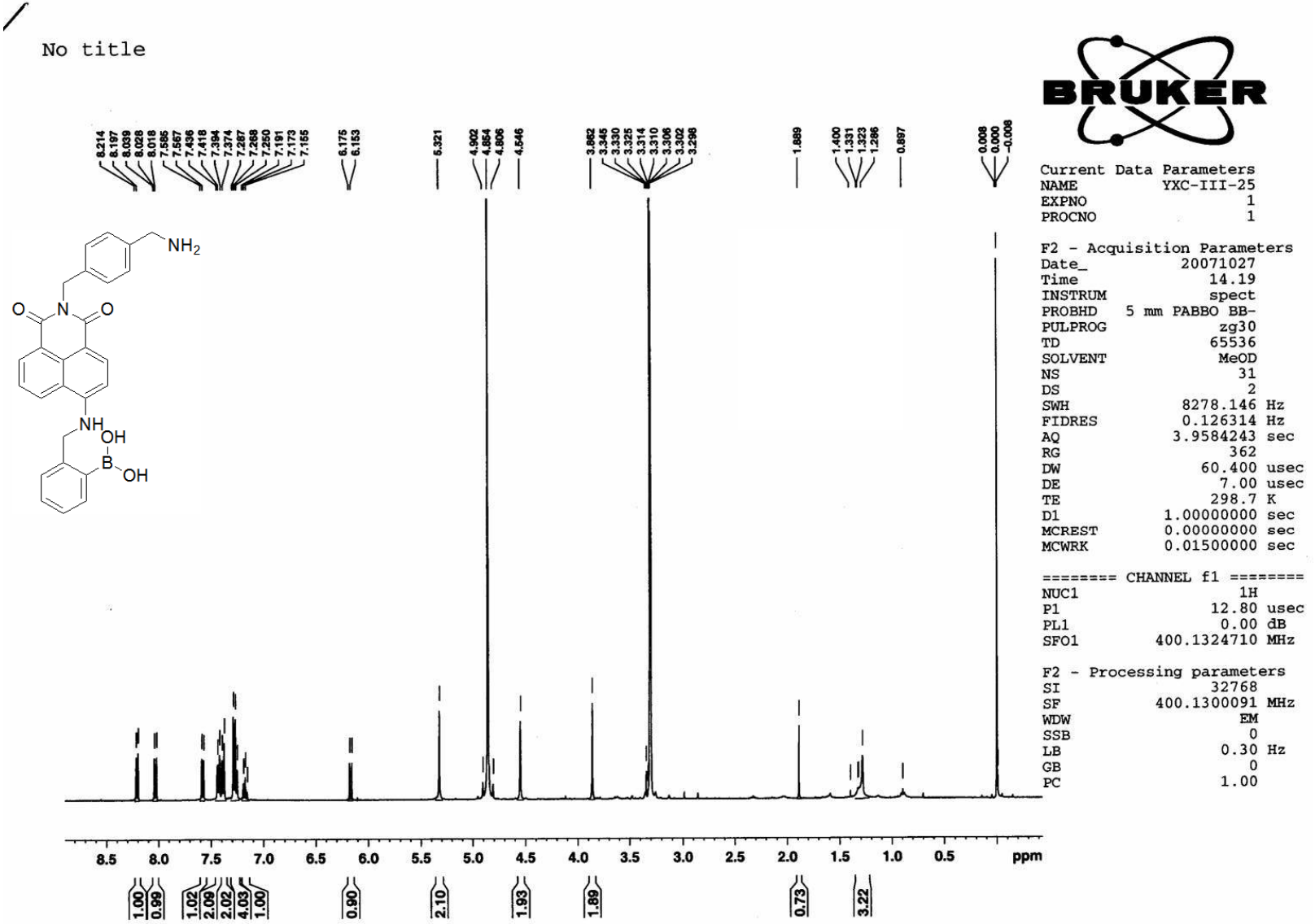
======== CHANNEL f1 ========
NUC1 13C
P1 8.00 usec
PL1 -3.00 dB
SFO1 100.6228298 MHz

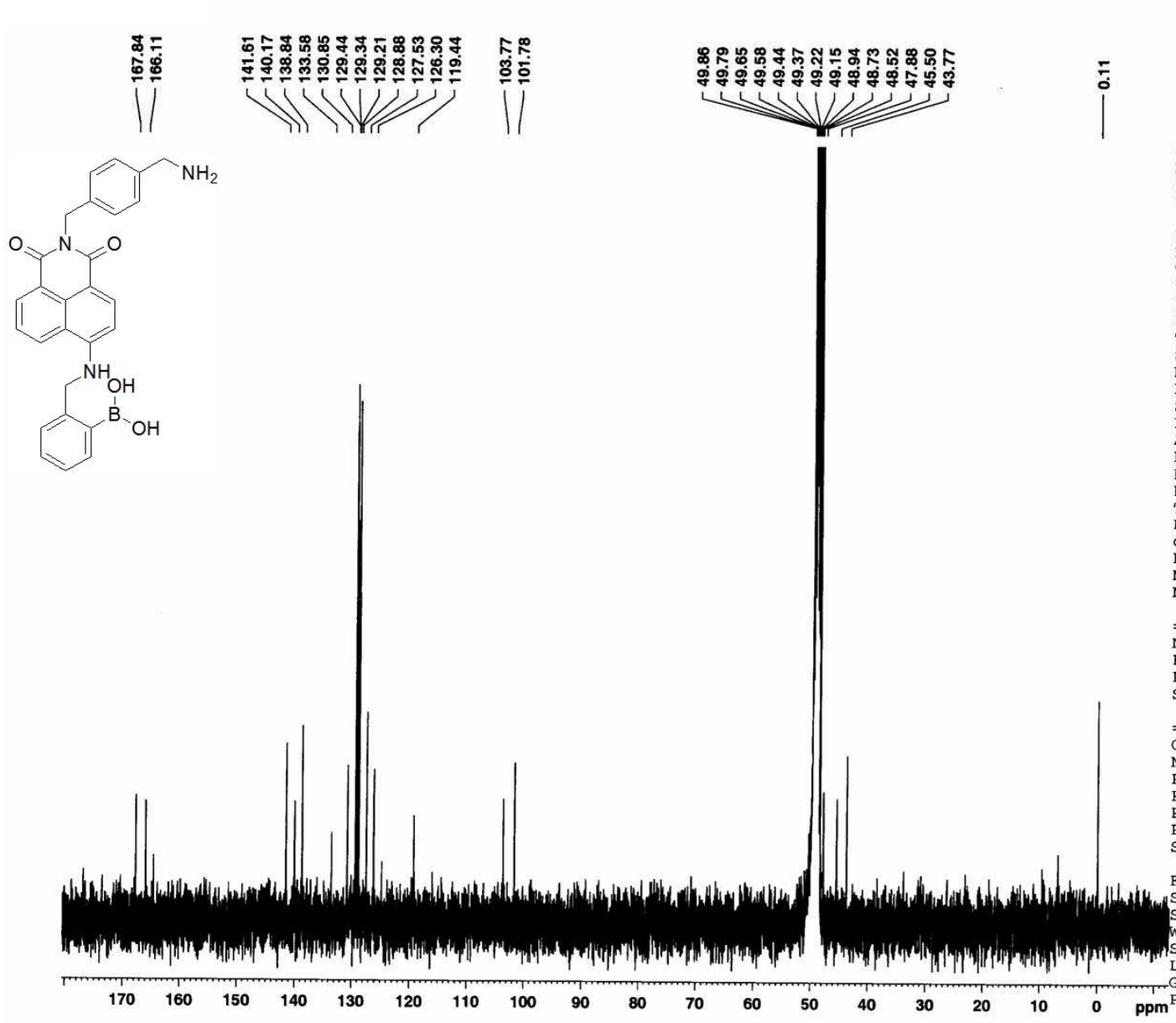
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 -1.00 dB
PL12 14.00 dB
PL13 14.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6126133 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


50%MeOH+0.5%NH3
15:57:3305-Jun
XIAOCHUANG_YXC_G_ESI_NEG_WANG_060509 44 (0.817) Cm (40:45)
1: TOF MS
100
%
0
564.3
578.3
592.4
546.3
536.3
608.3
622.3
642.3
463.2
260
280
300
320
340
360
380
400
420
440
460
480
500
520
540
560
580
600
620
640
660
680

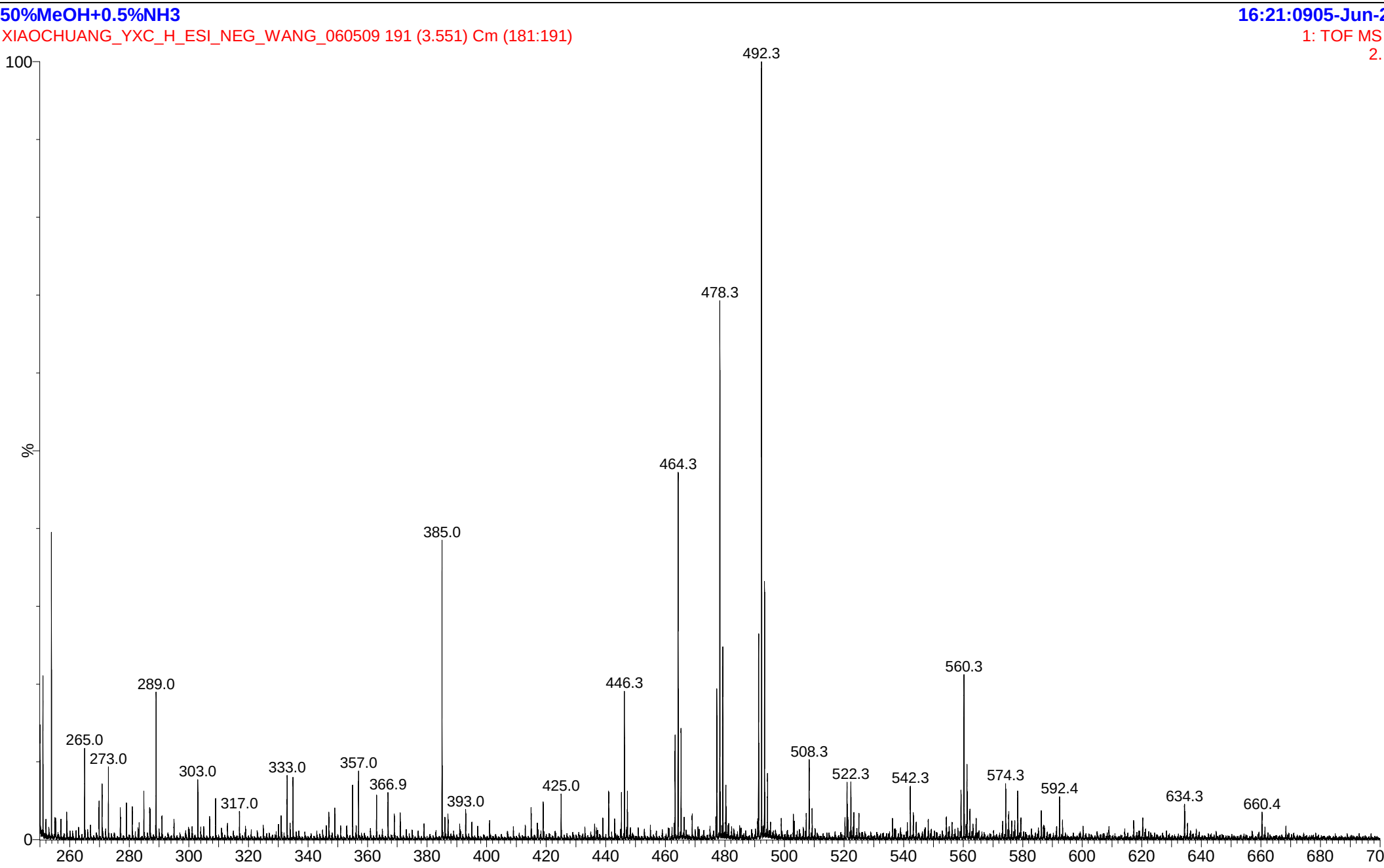

50%MeOH+0.5%NH3 LeuEnk as ITSD 554.2615 Da
16:00:1205-Jun-2009
XIAOCHUANG_YXC_G_ACCUMASS_ESI_NEG_WANG_060509 96 (1.782) AM (Cen,3, 80.00, Ar,6500.0,554.26,0.00); Cm (81:96)
1: TOF MS ES-
3.58e4
100
%
0
564.2310
578.2449
*
554.2615
592.2562
546.2229
608.2239
536.2195
622.2375
636.2438
500 505 510 515 520 525 530 535 540 545 550 555 560 565 570 575 580 585 590 595 600 605 610 615 620 625 630 635 640 645 650
m/z

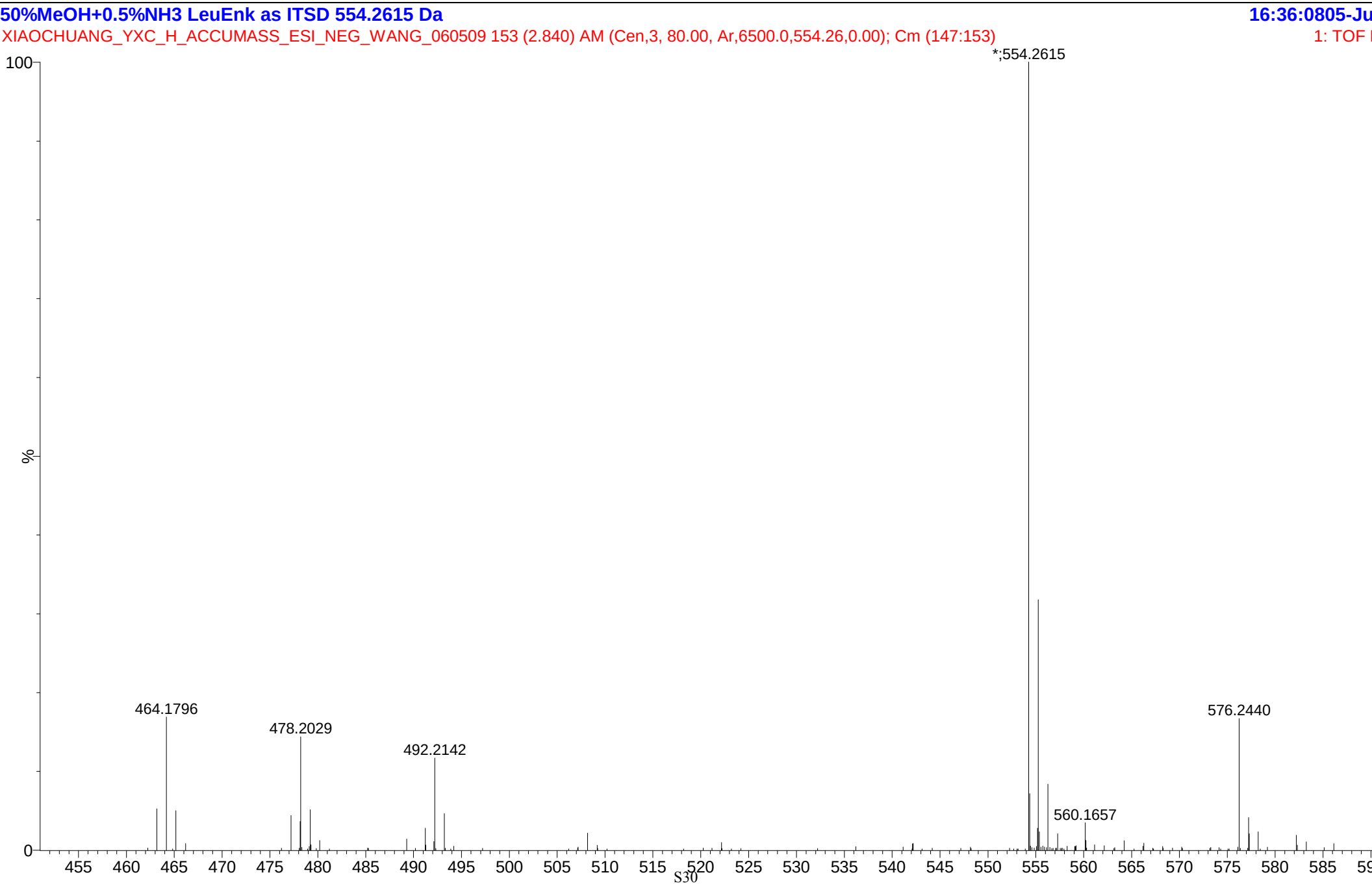

No title
BRUKER
8.214
8.197
8.039
8.026
8.018
7.585
7.567
7.436
7.418
7.394
7.374
7.287
7.268
7.250
7.191
7.173
7.155
6.175
6.153
5.321
4.902
4.854
4.806
4.646
3.862
3.345
3.330
3.325
3.314
3.310
3.306
3.302
3.298
1.889
1.400
1.331
1.323
1.286
0.897
0.008
0.000
-0.008
NH2
O
N
O
NH
OH
B
OH
8.5
8.0
7.5
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
ppm
1.00
0.99
1.02
2.09
2.02
4.03
1.00
0.90
2.10
1.93
1.89
0.73
3.22
Current Data Parameters
NAME YXC-III-25
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20071027
Time 14.19
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 31
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 362
DW 60.400 usec
DE 7.00 usec
TE 298.7 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 12.80 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300091 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

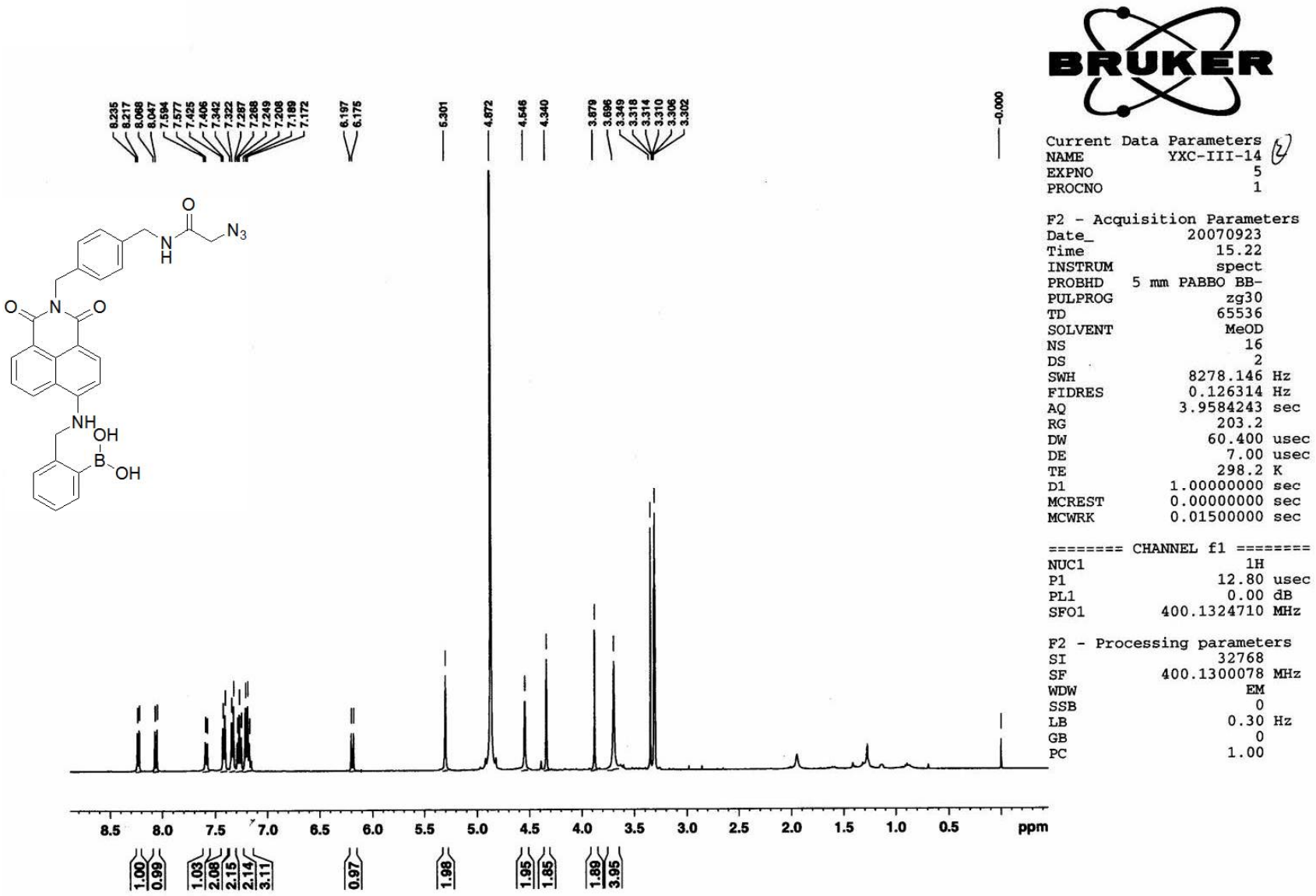


BRUKER

```
Current Data Parameters
NAME      YXC-III-25 C13
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20071027
Time              15.14
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG         zgpg30
TD               65536
SOLVENT            D2O
NS                5120
DS                   4
SWH          23980.814 Hz
FIDRES        0.365918 Hz
AQ            1.3664756 sec
RG               32768
DW              20.850 usec
DE               7.00 usec
TE              299.6 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                8.00 usec
PL1               -3.00 dB
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2            70.00 usec
PL2              -1.00 dB
PL12             14.00 dB
PL13             14.00 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6126118 MHz
WDW                  EM
SSB                   0
LB                1.00 Hz
GB                    0
PC                 1.40
```


50%MeOH+0.5%NH3
16:21:0905-Jun-2
XIAOCHUANG_YXC_H_ESI_NEG_WANG_060509 191 (3.551) Cm (181:191)
1: TOF MS
2.
100
%
0
492.3
478.3
464.3
385.0
446.3
289.0
265.0
273.0
303.0
317.0
333.0
357.0
366.9
393.0
425.0
508.3
522.3
542.3
560.3
574.3
592.4
634.3
660.4
260
280
300
320
340
360
380
400
420
440
460
480
500
520
540
560
580
600
620
640
660
680


50%MeOH+0.5%NH3 LeuEnk as ITSD 554.2615 Da
16:36:0805-Ju
XIAOCHUANG_YXC_H_ACCUMASS_ESI_NEG_WANG_060509 153 (2.840) AM (Cen,3, 80.00, Ar,6500.0,554.26,0.00); Cm (147:153)
1: TOF
*;554.2615
100
%
0
464.1796
478.2029
492.2142
560.1657
576.2440
455 460 465 470 475 480 485 490 495 500 505 510 515 520 525 530 535 540 545 550 555 560 565 570 575 580 585 59



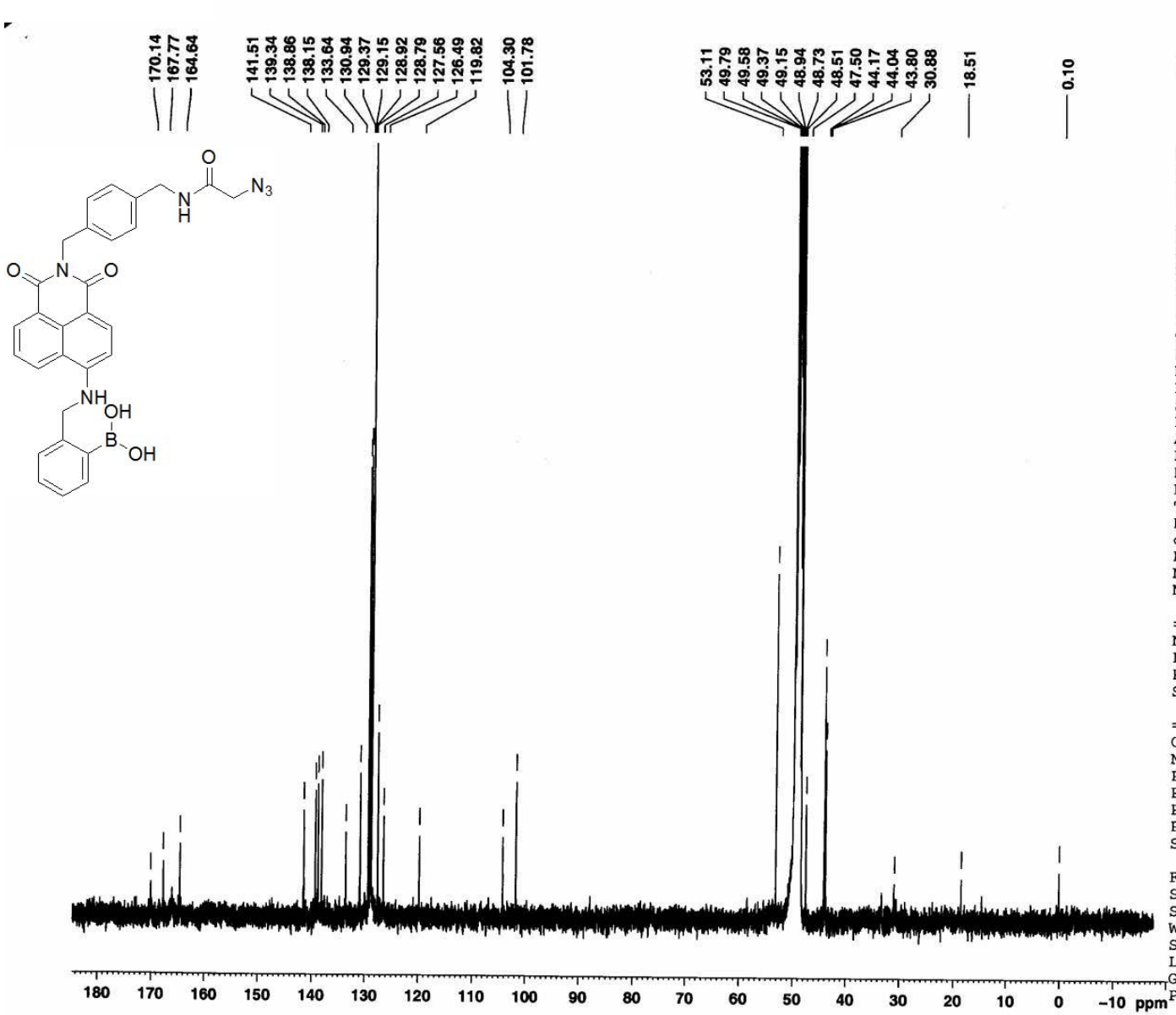
BRUKER

Current Data Parameters
NAME YXC-III-14
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070923
Time 15.22
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 203.2
DW 60.400 usec
DE 7.00 usec
TE 298.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 12.80 usec
PL1 0.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300078 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



BRUKER

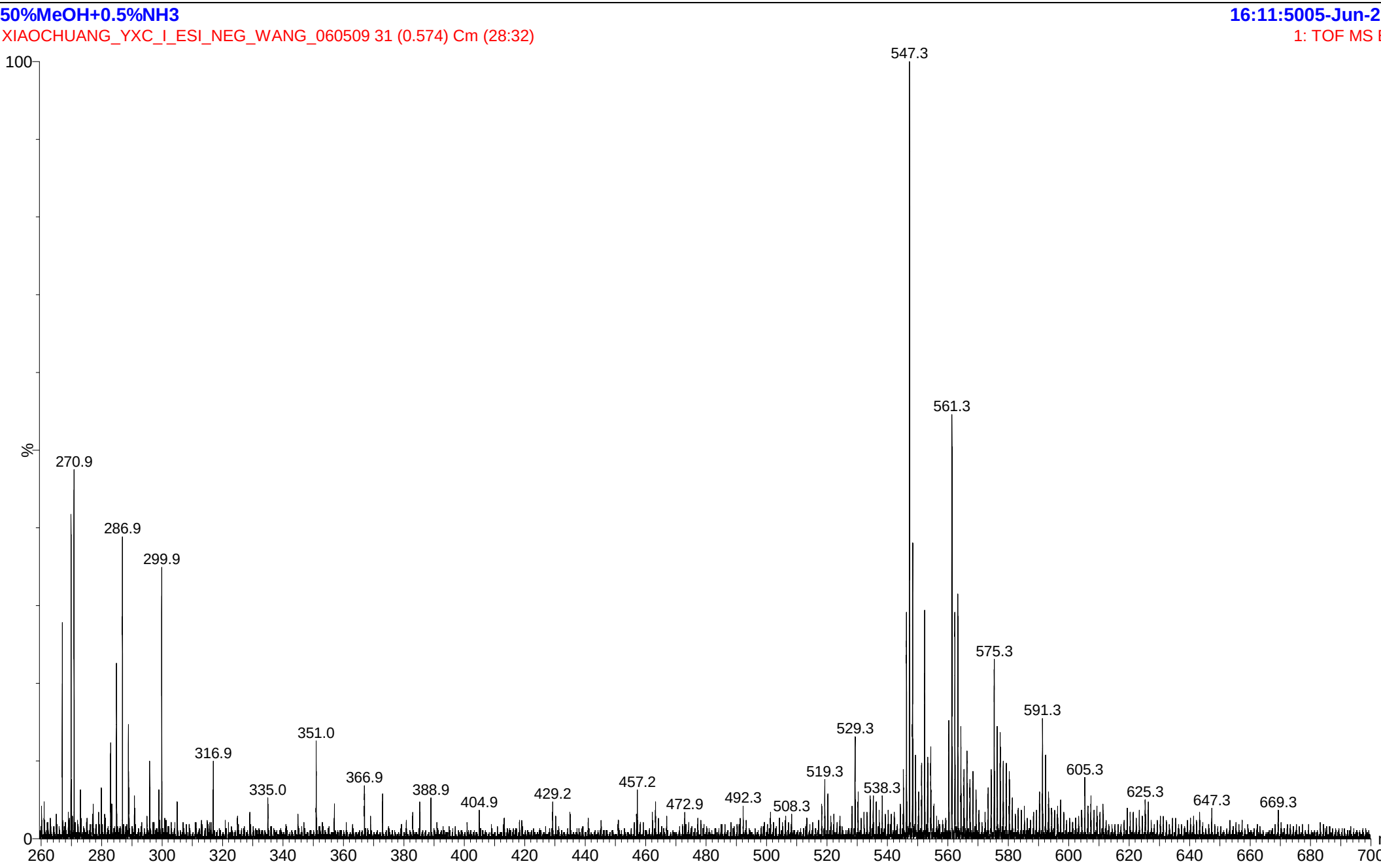
Current Data Parameters
NAME YXC-III-14 C13
EXPNO 5
PROCNO 1

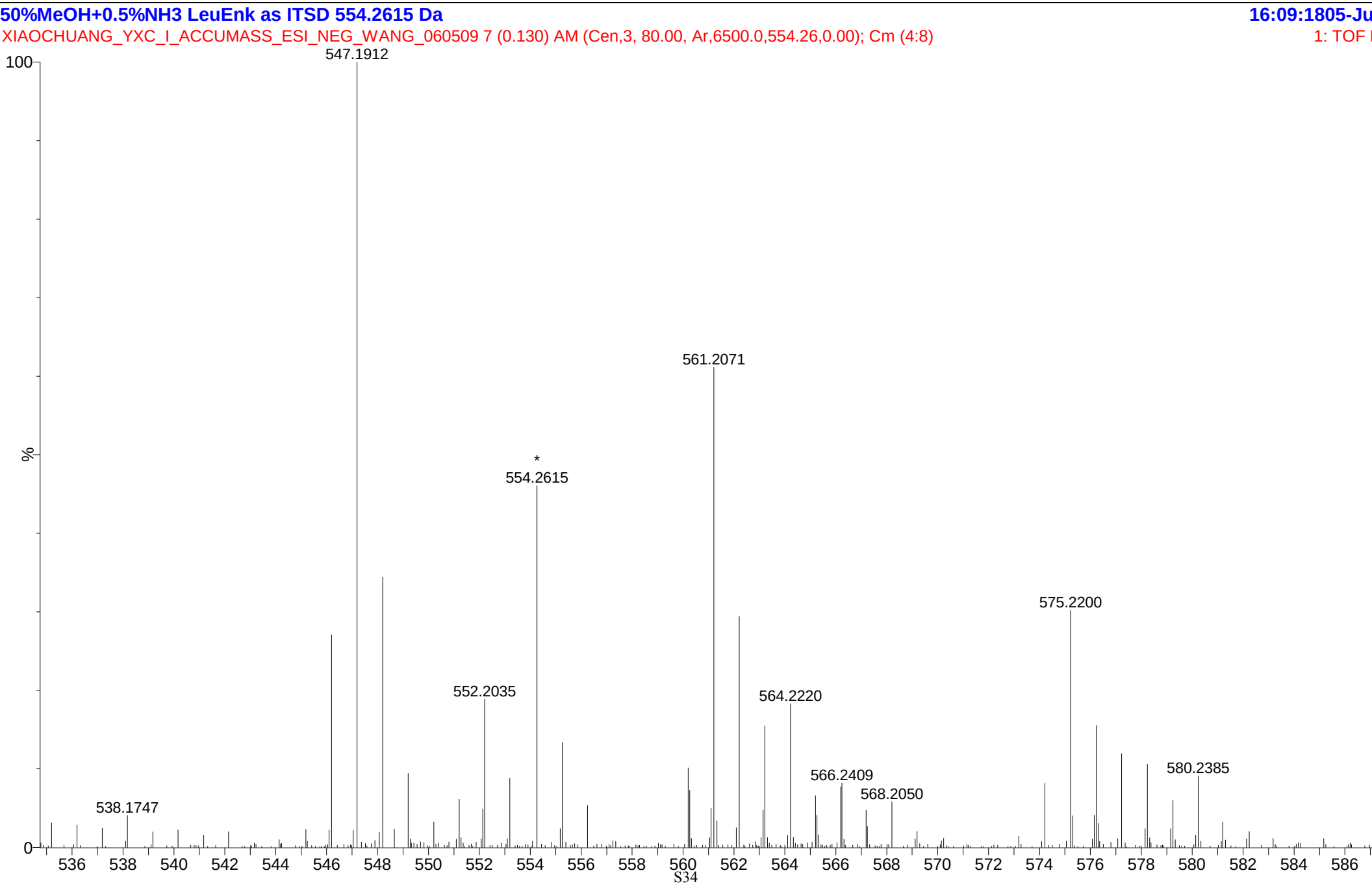
F2 - Acquisition Parameters
Date_ 20070921
Time 22.15
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT D2O
NS 12000
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 32768
DW 20.850 usec
DE 7.00 usec
TE 298.4 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

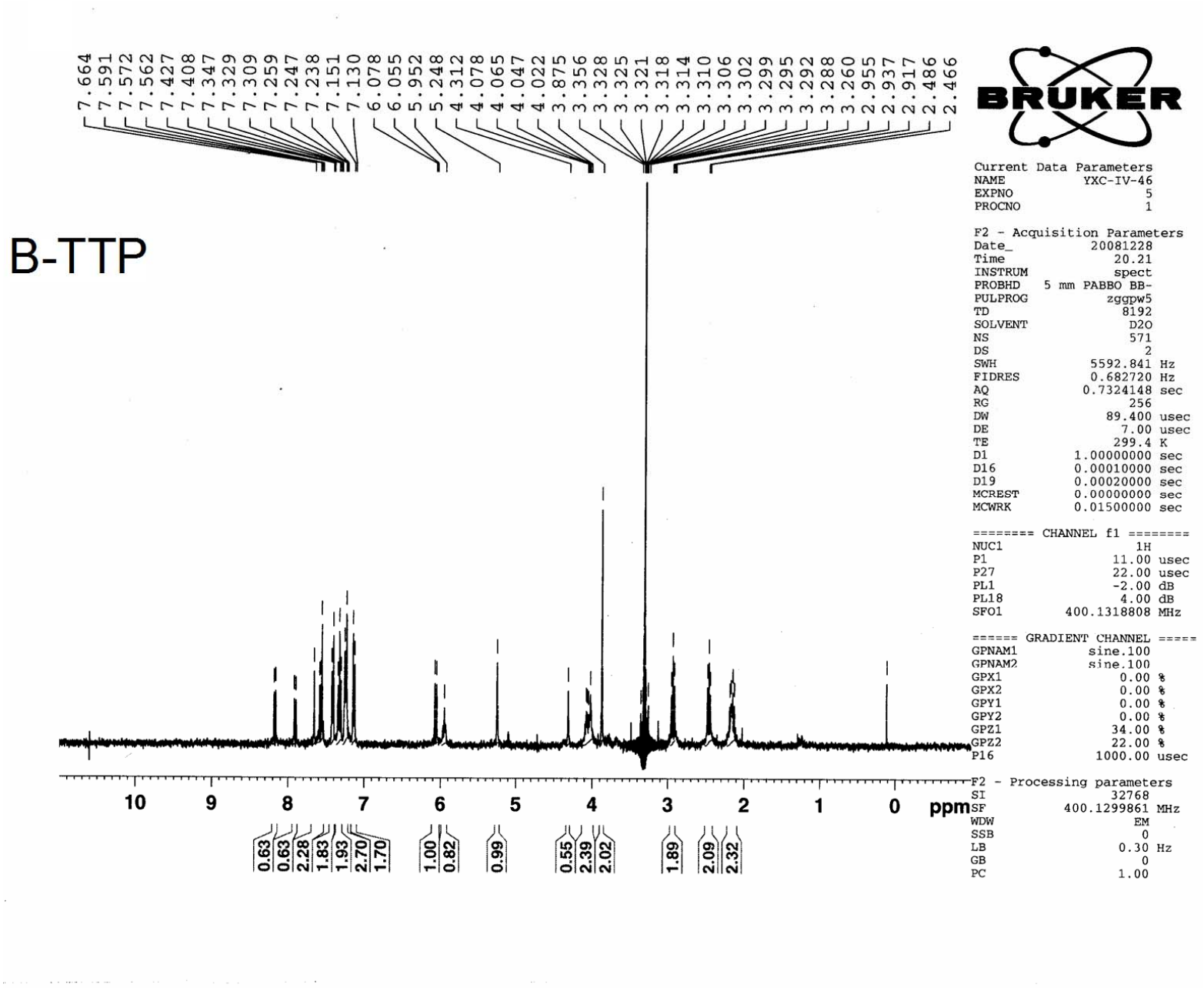
======== CHANNEL f1 ========
NUC1 13C
P1 8.00 usec
PL1 -3.00 dB
SFO1 100.6228298 MHz

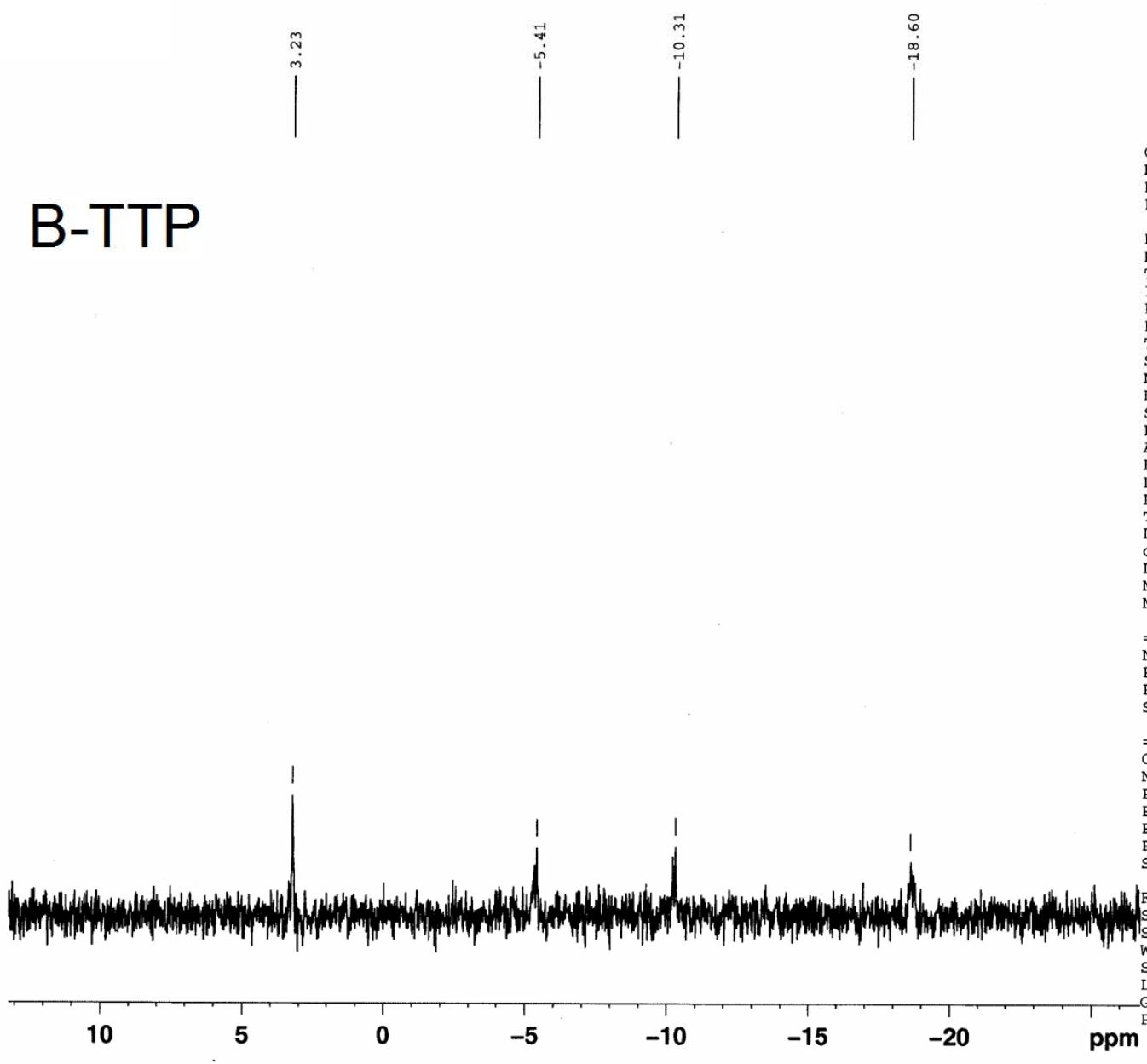
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 -1.00 dB
PL12 14.00 dB
PL13 14.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6126140 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

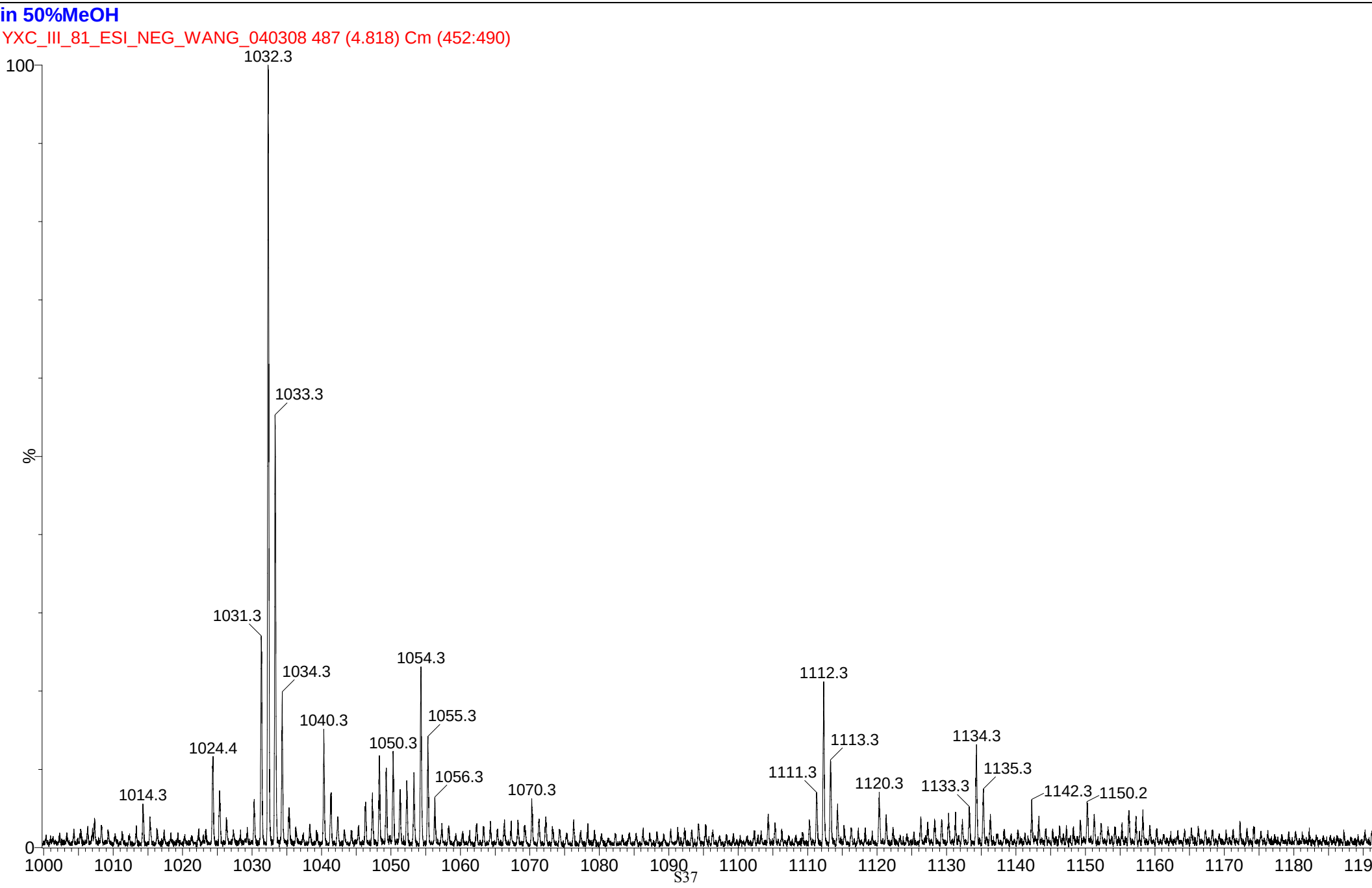

50%MeOH+0.5%NH3
16:11:5005-Jun-2
XIAOCHUANG_YXC_I_ESI_NEG_WANG_060509 31 (0.574) Cm (28:32)
1: TOF MS E
100
%
0
547.3
561.3
270.9
286.9
299.9
316.9
351.0
335.0
366.9
388.9
404.9
429.2
457.2
472.9
492.3
508.3
519.3
529.3
538.3
575.3
591.3
605.3
625.3
647.3
669.3
260
280
300
320
340
360
380
400
420
440
460
480
500
520
540
560
580
600
620
640
660
680
700


50%MeOH+0.5%NH3 LeuEnk as ITSD 554.2615 Da
16:09:1805-Ju
XIAOCHUANG_YXC_I_ACCUMASS_ESI_NEG_WANG_060509 7 (0.130) AM (Cen,3, 80.00, Ar,6500.0,554.26,0.00); Cm (4:8)
1: TOF
547.1912
561.2071
*
554.2615
575.2200
552.2035
564.2220
580.2385
566.2409
568.2050
538.1747
100
%
0
536 538 540 542 544 546 548 550 552 554 556 558 560 562 564 566 568 570 572 574 576 578 580 582 584 586


B-TTP
7.664
7.591
7.572
7.562
7.427
7.408
7.347
7.329
7.309
7.259
7.247
7.238
7.151
7.130
6.078
6.055
5.952
5.248
4.312
4.078
4.065
4.047
4.022
3.875
3.356
3.328
3.325
3.321
3.318
3.314
3.310
3.306
3.302
3.299
3.295
3.292
3.288
3.260
2.955
2.937
2.917
2.486
2.466
10
9
8
7
6
5
4
3
2
1
0
ppm
0.63
0.63
2.28
1.83
1.93
2.70
1.70
1.00
0.82
0.99
0.55
2.39
2.02
1.89
2.09
2.32
BRUKER
Current Data Parameters
NAME YXC-IV-46
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081228
Time 20.21
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zggpw5
TD 8192
SOLVENT D2O
NS 571
DS 2
SWH 5592.841 Hz
FIDRES 0.682720 Hz
AQ 0.7324148 sec
RG 256
DW 89.400 usec
DE 7.00 usec
TE 299.4 K
D1 1.00000000 sec
D16 0.00010000 sec
D19 0.00020000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
P27 22.00 usec
PL1 -2.00 dB
PL18 4.00 dB
SFO1 400.1318808 MHz
====== GRADIENT CHANNEL =====
GPNAM1 sine.100
GPNAM2 sine.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 34.00 %
GPZ2 22.00 %
P16 1000.00 usec
F2 - Processing parameters
SI 32768
SF 400.1299861 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00




```
Current Data Parameters
NAME       YXC-IV-46 31P
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20081228
Time              16.59
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT             D2O
NS                 3872
DS                    4
SWH           64935.066 Hz
FIDRES         0.990830 Hz
AQ             0.5046772 sec
RG               20642.5
DW                7.700 usec
DE                7.00 usec
TE                299.5 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 31P
P1                  7.50 usec
PL1                -2.00 dB
SFO1        161.9674742 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              70.00 usec
PL2                -1.00 dB
PL12               14.00 dB
PL13               14.00 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                  32768
SF          161.9755066 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```


in 50%MeOH
YXC_III_81_ESI_NEG_WANG_040308 487 (4.818) Cm (452:490)
100
%
0
1032.3
1033.3
1031.3
1034.3
1054.3
1055.3
1040.3
1050.3
1024.4
1056.3
1014.3
1070.3
1112.3
1113.3
1111.3
1120.3
1134.3
1135.3
1133.3
1142.3
1150.2
1000
1010
1020
1030
1040
1050
1060
1070
1080
1090
1100
1110
1120
1130
1140
1150
1160
1170
1180
119

50%MeOH+50%H2O+0.5%NH3, Naformate as standard **16:20:0011-Aug-2009**

XIAOCHUAN_YXC_N_TTP_ESI_NEG_WANG_081109_04 194 (3.598) AM (Cen,3, 80.00, Ar,6500.0,1064.81,0.00); Sm (SG, 2x3.00); Cm (97:199) 1: TOF MS ES- 3.23e3