

Biological screening of a diverse set of AI-2 analogues in *Vibrio harveyi* suggests that receptors which are involved in synergistic agonism of AI-2 and analogues are promiscuous.

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General Methods of Synthesis

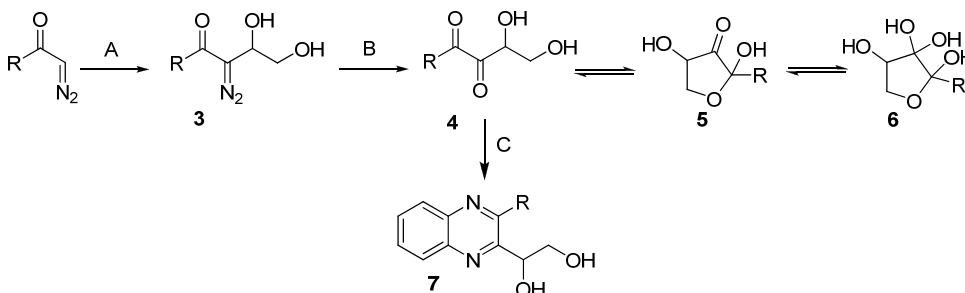
Air and moisture sensitive reactions were carried out in oven-dried glasswares sealed with rubber septa under a positive pressure of dry argon or nitrogen, unless otherwise indicated. Reactions were stirred using Teflon-coated magnetic stir bars. Organic solutions were concentrated using a Büchi rotary evaporator with an aspirator pump. Dry tetrahydrofuran was obtained using a solvent purification system (PureSolvent™) prior to use. Dry acetonitrile was distilled from CaH₂ prior to use. Thin-layer chromatography (TLC) was performed on Merck Kieselgel 60 F254 plates with a 365 nm fluorescent indicator. The TLC was visualized by ultraviolet light and acidic *p*-anisaldehyde stain followed by gentle heating. The crude reaction mixtures were purified by flash chromatography on silica gel (230-400 mesh).

NMR spectra were measured on Bruker AV-400, Bruker DRX-400 (¹H at 400 MHz, ¹³C at 100MHz), Bruker DRX-500 (¹H at 500 MHz, ¹³C at 125MHz) or Bruker AVIII-600 (¹H at 600 MHz, ¹³C at 150MHz). Data for ¹H -NMR spectra are reported as follows: chemical shift (ppm, relative to residual solvent peaks or indicated external standards; s = singlet, d = doublet, t = triplet, q = quartet, dd = doublet of doublets, td = triplet of doublets, ddd = doublet of doublet of doublets, dddd = doublet of doublet of doublet of doublets, m = multiplet), coupling constant (Hz), and integration. Data for ¹³C -NMR are reported in terms of chemical shift (ppm) relative to residual solvent peak. Mass spectra (MS) and high resolution mass spectra (HRMS) were recorded by JEOL AccuTOF-CS (ESI positive, needle voltage 1000 eV). Infrared spectra (IR) were recorded by a ThermoNicolet IR200 Spectrometer

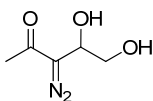
Bioluminescence assay

V. harveyi strain MM32 was obtained from ATCC. All bacteria were maintained on AB agar at 30°C and grown in AB media at 30°C with 250rpm. Overnight culture of MM32 were diluted 1:5000 in fresh AB media with 100µM of boric acid and AI-2 and/or analogues at the indicated

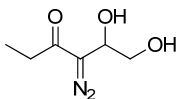
concentration and grown for 8 hours at 30 °C with 250rpm. At the 8 hour time point, bacteria for all samples have reached an OD₆₀₀ of 0.6-0.8. The luminescence of eight independent cultures were determined for each analogue using a Spectromax M5 (Molecular Devices). All samples are reported as averages of the readings with error as one standard deviation from the mean.



A: To a solution of the diazocarbonyl in anhydrous acetonitrile (0.2 M) was added DBU (0.16-0.20 eq.) and 2-(*tert*-butyldimethylsilyloxy)acetaldehyde (1.0-1.5 eq.).¹ The reaction was stirred at room temperature under nitrogen for 4-8 hours and monitored by TLC. Upon the disappearance of starting material, the reaction was quenched with a saturated solution of sodium bicarbonate. The organic layer was extracted with dichloromethane (3 x 20 mL) and dried with magnesium sulfate. The solvent was evaporated under reduced pressure. To a solution of crude product in anhydrous tetrahydrofuran (0.2 M) was added TBAF (1-2 eq.) at 0 °C. The solution was allowed to warm to room temperature and stirred for 1-3 hours under nitrogen. The solvent was evaporated and the crude product was purified by column chromatography. The product eluted with 1:3 to 3:2 ethyl acetate: hexane. Compound 3 was eluted as yellow oil (20-52 % yield over 2 steps).

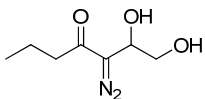


3-diazo-4,5-dihydroxypentan-2-one (3a): ¹H NMR (400 MHz, CDCl₃) δ ppm 4.76 (1H, m), 3.85 (1H, dd, *J* = 11.4, 3.2 Hz), 3.75 (1H, dd, *J* = 11.4, 3.2 Hz), 3.46 (1H, s, br), 2.69 (1H, s, br), 2.26 (3H, s). ¹³C NMR (100 MHz, CDCl₃) δ ppm 191.7, 66.3, 64.2, 25.6. IR: 3349, 2361, 2338, 2092, 1607 cm⁻¹. Yield: 50% (over 2 steps)

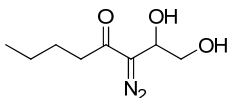


4-diazo-5,6-dihydroxyhexan-3-one (3b): ¹H NMR (400 MHz, CDCl₃) δ ppm 4.75 (1H, m), 4.48 (1H, br), 4.21 (1H, br), 3.81-3.79 (1H, m), 3.72-3.70 (1H, m), 3.44 (1H, br), 2.50 (2H, q, *J* = 7.4

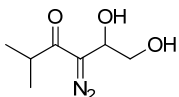
Hz), 1.13 (3H, t, $J = 7.4$ Hz); ^{13}C NMR (100 MHz, CDCl_3) δ ppm 195.2, 65.9, 64.1, 31.2, 8.2; IR: 3365, 2980, 2940, 2084, 1607 cm^{-1} . Yield 33% (over 2 steps)



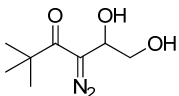
3-diazo-1,2-dihydroxyheptan-4-one (3c): ^1H NMR (400 MHz, CDCl_3) δ ppm 4.75 (1H, m), 4.08 (1H, br), 3.81 (1H, dd, $J = 11.2, 3.4$ Hz), 3.70 (1H, dd, $J = 11.2, 5.4$ Hz), 3.35 (1H, br), 2.45 (2H, t, $J = 7.4$ Hz), 1.68-1.63 (2H, m), 0.94 (3H, t, $J = 7.4$ Hz). ^{13}C NMR (100 MHz, CDCl_3) δ ppm 195.3, 66.6, 64.8, 40.5, 18.6, 14.1. IR: 3395, 2964, 2935, 2876, 2082, 1605 cm^{-1} . Yield: 52% (over 2 steps)



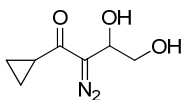
3-diazo-1,2-dihydroxyoctan-4-one (3d): ^1H NMR (400 MHz, CDCl_3) 4.75 (1H, br), 3.83 (1H, dd, $J = 3.9, 11.4$ Hz), 3.72 (1H, dd, $J = 5.3, 11.5$ Hz), 3.02 (1H, s, br), 2.45-2.49 (2H, m), 1.95 (1H, s, br), 1.57-1.65 (2H, m), 1.30-1.40 (2H, m), 0.91 (3H, t, $J = 7.3$ Hz) ^{13}C NMR (100 MHz, CDCl_3) δ ppm 194.9, 66.2, 64.1, 37.7, 26.5, 22.1, 13.6. IR: 3334, 2959, 2872, 2082, 1607 cm^{-1} . Yield: 48% (over 2 steps)



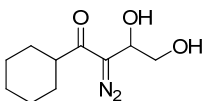
4-diazo-5,6-dihydroxy-2-methylhexan-3-one (3e): ^1H NMR (400 MHz, CDCl_3) δ ppm 4.74-4.76 (1H, m), 3.81 (1H, dd, $J = 11.4, 3.9$ Hz), 3.70 (1H, dd, $J = 11.5, 5.6$ Hz), 2.79-2.86 (1H, m), 1.12 (6H, d, $J = 6.8$ Hz). ^{13}C NMR (100 MHz, CDCl_3) δ ppm 199.5, 66.7, 64.8, 36.5, 19.1. IR: 3357, 2971, 2929, 2362, 2084, 1738, 1609 cm^{-1} . Yield: 25% (over 2 steps)



4-diazo-5,6-dihydroxy-2,2-dimethylhexan-3-one (3f): ^1H NMR (400 MHz, CDCl_3) δ ppm 4.78 (1H, t, $J = 4.9$ Hz), 3.83 (1H, dd, $J = 4.2, 11.5$ Hz), 3.74-3.70 (1H, m), 1.23 (9H, s). ^{13}C NMR (100 MHz, CDCl_3) δ ppm 200.6, 67.8, 64.0, 44.3, 26.6. IR: 3358, 2971, 2361, 2338, 2077, 1702, 1602 cm^{-1} . Yield: 20% (over 2 steps)

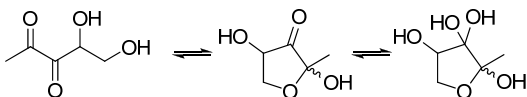


1-cyclopropyl-2-diazo-3,4-dihydroxybutan-1-one (3g): ^1H NMR (400 MHz, CDCl_3) δ ppm 4.81 (1H, s, br), 3.87-3.92 (1H, m), 3.79-3.83 (1H, m), 3.38 (1H, s, br), 2.62 (1H, s, br), 1.94-2.01 (1H, m), 1.13-1.17 (2H, m), 0.92-0.97 (2H, m); ^{13}C NMR (100 MHz, CDCl_3) δ ppm 193.5, 66.4, 63.7, 16.4, 9.5. IR: 3401, 2929, 2362, 2088, 1690, 1612 cm^{-1} . Yield: 28% (over 2 steps)

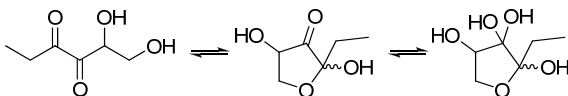


1-cyclohexyl-2-diazo-3,4-dihydroxybutan-1-one (3h): ^1H NMR (400 MHz, CDCl_3) δ ppm 4.75 (1H, t, $J = 4.6, 4.6\text{Hz}$), 3.85 (1H, dd, $J = 4.1, 11.4\text{Hz}$), 3.75 (1H, dd, $J = 4.9, 11.5\text{Hz}$), 3.41 (1H, s, br), 2.58 (1H, s, br), 2.49-2.56 (1H, m), 1.74-1.83 (3H, m), 1.68-1.70 (1H, m), 1.42-1.51 (2H, m), 1.22-1.32 (3H, m). ^{13}C NMR (100 MHz, CDCl_3) δ ppm 198.4, 66.7, 64.3, 46.3, 28.8, 25.5. IR: 3399, 2975, 2932, 2362, 2085, 1616 cm^{-1} . Yield: 36% (over 2 steps)

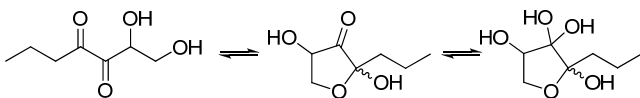
B: To a solution of Compound **3** (1 eq.) in acetone (1-2 mL) was added dioxirane (15-20 mL) in acetone dropwise. The reaction was allowed to stir at room temperature (1-2 hrs) until complete disappearance of starting material as indicated by TLC (loss of UV activity). Solvent and excess reagent was evaporated under reduced pressure. NMR was taken without further purification.



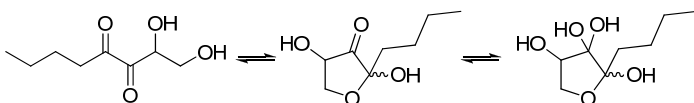
Equilibrium mixture of compounds 4a-6a: ^1H NMR (400 MHz, D_2O) δ ppm 4.24-4.28 (m), 4.04-4.10 (m), 3.93-3.95 (m), 3.84-3.86 (m), 3.67-3.72 (m), 3.52-3.59 (m), 3.44-3.48 (m), 2.26 (s), 1.30 (s), 1.26 (s). ^{13}C NMR (100 MHz, D_2O) δ ppm 103.9, 99.1, 74.3, 73.5, 71.2, 61.4, 24.8, 20.2, 19.6.



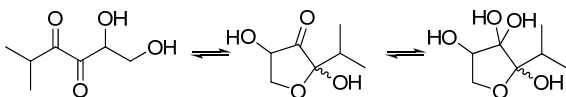
Equilibrium mixture of compounds 4b-6b: ^1H NMR (400 MHz, CDCl_3) δ ppm 4.94 (t, $J = 3.1$), 3.99-4.07 (m), 2.91-3.01 (1H, m), 2.75-2.85 (1H, m), 1.80-1.88 (2H, m), 1.15 (t, $J = 7.2\text{ Hz}$). ^{13}C NMR (100 MHz, CDCl_3) δ ppm 200.2, 198.8, 75.2, 64.2, 30.9, 18.9, 6.9.



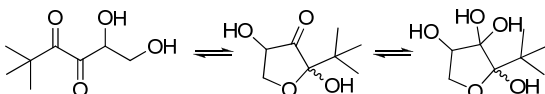
Equilibrium mixture of compounds 4c-6c: ^1H NMR (400 MHz, CDCl_3) δ ppm 4.91 (t, $J = 3.1$ Hz), 3.96-4.04 (m), 2.81-2.89 (m), 2.70-2.78 (m), 1.61-1.70 (m), 0.88-0.99 (m). ^{13}C NMR (100 MHz, CDCl_3) δ ppm 199.3, 198.4, 74.7, 63.7, 38.7, 16.2, 13.5.



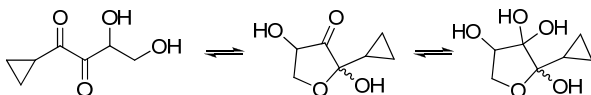
Equilibrium mixture of compounds 4d-6d: ^1H NMR (400 MHz, CDCl_3) δ ppm 4.94, (t, $J = 3.2$ Hz), 3.98-4.06 (m), 2.86-2.94 (m), 2.74-2.83 (m), 1.59-1.67 (m), 1.34-1.43 (m), 0.95 (t, $J = 7.3$ Hz). ^{13}C NMR (400 MHz, CDCl_3) δ ppm 199.2, 198.2, 74.5, 63.5, 36.4, 24.4, 21.9, 13.5.



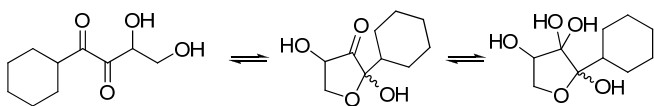
Equilibrium mixture of compounds 4e-6e: ^1H NMR (400 MHz, CDCl_3) δ ppm 4.92 (t, $J = 3.2$ Hz), 3.98 (d, $J = 3.3$ Hz), 3.72 (q, $J = 7.0$ Hz), 3.34-3.41 (m), 1.23-1.29 (m), 1.19 (dd, $J = 6.9, 1.4$ Hz), 1.15 (dd, $J = 6.9, 6.3$ Hz), 1.03 (dd, $J = 6.9, 1.5$ Hz), 0.92 (d, $J = 6.9$ Hz); ^{13}C NMR (100 MHz, CDCl_3) δ ppm 203.1, 199.5, 75.3, 63.9, 35.3, 34.1, 30.1, 17.7, 17.3.



Equilibrium mixture of compounds 4f-6f: ^1H NMR (400 MHz, CDCl_3) δ ppm 4.78 (t, $J = 3.5$ Hz), 4.37-4.45 (m), 3.91 (d, $J = 3.5$ Hz), 3.69-3.74 (m), 1.27 (s), 1.23 (s), 1.09 (s), 1.06 (s), 1.01 (s). ^{13}C NMR (100 MHz, CDCl_3) δ ppm 213.8, 207.4, 201.4, 101.7, 75.5, 73.1, 66.8, 63.2, 42.9, 37.1, 26.6, 26.1, 24.6, 24.1.

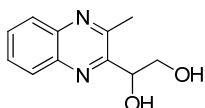


Equilibrium mixture of compounds 4g-6g: ^1H NMR (400 MHz, CDCl_3) δ ppm 4.90 (t, $J = 3.2$ Hz), 4.00 (d, $J = 3.2$ Hz), 2.71-2.75 (m), 1.09-1.24 (m), 0.83-0.89 (m). ^{13}C NMR (100 MHz, CDCl_3) δ ppm 74.7, 63.7, 29.7, 16.3, 14.3.

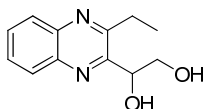


Equilibrium mixture of compounds 4g-6g: ^1H NMR (400 MHz, CDCl_3) δ ppm 4.93 (br), 4.00 (d, $J = 2.8$ Hz), 3.14-3.20 (m), 1.71-1.92 (m), 1.23-1.48 (m). ^{13}C NMR (100 MHz, CDCl_3) δ ppm 202.4, 199.6, 75.2, 63.9, 44.6, 28.1, 27.6, 26.1, 26.0, 25.9, 25.5.

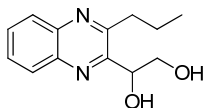
C: To a solution of DPD-analogues was added 1,2-phenylenediamine (1.5 eq.). The reaction was stirred at room temperature for 10 minutes and then the reaction mixture was washed with 2 M HCl aqueous solution. The crude mixture was purified with silica-gel column chromatography (hexane: ethyl acetate).



1-(3-methylquinoxalin-2-yl)ethane-1,2-diol (7a): ^1H NMR (500 MHz, CDCl_3) δ ppm 8.03-8.06 (2H, m), 7.72-7.78 (2H, m), 5.11-5.16 (1H, m, br), 4.55 (1H, d, $J = 7.6$ Hz), 4.03-4.08 (1H, m), 3.86 (1H, dd, $J = 11.4, 5.5$ Hz), 2.83 (3H, s). ^{13}C NMR (125 MHz, CDCl_3) δ ppm 153.1, 152.2, 142.1, 139.6, 130.3, 129.8, 128.8, 128.6, 71.2, 66.2, 29.9, 22.2. HRMS (ESI⁺): Found 205.0978 Calc'd 205.0977 (M+H).

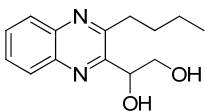


1-(3-ethylquinoxalin-2-yl)ethane-1,2-diol (7b): ^1H NMR (500 MHz, CDCl_3) δ ppm 8.10 (1H, dd, $J = 8.4, 1.8$ Hz), 8.02 (1H, d, $J = 8.0$ Hz), 7.71-7.78 (2H, m), 5.17 (1H, s, br), 4.56 (1H, d, $J = 7.0$ Hz), 4.01-4.06 (1H, m), 3.83 (1H, dd, $J = 11.6, 5.7$ Hz), 3.02-3.16 (2H, m), 1.46 (3H, t, $J = 7.5$ Hz). ^{13}C NMR (125 MHz, CDCl_3) δ ppm 156.4, 152.7, 142.3, 139.5, 130.2, 129.7, 128.9, 128.6, 70.9, 66.7, 29.6, 12.8. HRMS (ESI⁺): Found 219.1131 Calc'd 219.1134 (M+H).

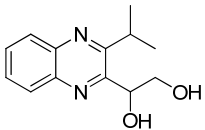


1-(3-propylquinoxalin-2-yl)ethane-1,2-diol (7c): ^1H NMR (500 MHz, CDCl_3) δ ppm 8.06 (1H, dd, $J = 8.3, 1.7$ Hz), 8.02 (1H, d, $J = 8.0$ Hz), 7.70-7.77 (2H, m), 5.17 (1H, s, br), 4.56 (1H, d, $J = 7.0$ Hz), 4.06-4.01 (1H, m), 3.83 (1H, dd, $J = 11.7, 5.7$ Hz), 3.02-3.16 (2H, m), 1.46 (3H, t, $J =$

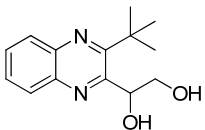
7.5 Hz). ^{13}C NMR (125 MHz, CDCl_3) δ ppm 156.4, 152.7, 142.3, 139.5, 130.2, 129.7, 128.9, 128.6, 70.9, 66.7, 29.6, 12.8. HRMS (ESI+): Found 233.1331 Calc'd 233.1290 (M+H).



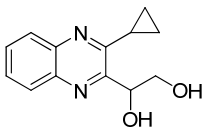
1-(3-butylquinoxalin-2-yl)ethane-1,2-diol (7d): ^1H NMR (500 MHz, CDCl_3) δ ppm 8.07 (1H, d, J = 8.0 Hz), 8.03 (1H, d, J = 7.5 Hz), 7.71-7.77 (2H, m), 5.17 (1H, s, br), 4.57 (1H, s, br), 4.04 (1H, dd, J = 11.6, 3.2 Hz), 3.81 (1H, dd, J = 11.6, 5.9 Hz), 2.99-3.10 (2H, m), 1.79-1.94 (2H, m), 1.47-1.55 (2H, m), 1.00 (3H, t, J = 7.4 Hz). ^{13}C NMR (125 MHz, CDCl_3) δ ppm 155.8, 152.8, 142.3, 139.4, 130.2, 129.7, 128.9, 128.6, 70.9, 66.8, 34.3, 31.2, 23.1, 14.2. HRMS (ESI+): Found 247.1459 Calc'd 247.1447 (M+H).



1-(3-isopropylquinoxalin-2-yl)ethane-1,2-diol (7e): ^1H NMR (600 MHz, CDCl_3) δ ppm 8.08 (1H, d, J = 7.8 Hz), 8.03 (1H, d, J = 7.8 Hz), 7.71-7.77 (2H, m), 5.22 (1H, s), 4.69 (1H, s), 4.03 (1H, dd, J = 11.4, 2.5 Hz), 3.77 (1H, dd, J = 11.6, 6.0 Hz), 3.43-3.47 (1H, m), 1.45 (3H, d, J = 6.7 Hz), 1.39 (3H, d, J = 6.7 Hz). ^{13}C NMR (150 MHz, CDCl_3) δ ppm 160.4, 151.9, 142.5, 139.4, 130.0, 129.7, 129.1, 128.5, 70.7, 67.2, 31.2, 29.9, 22.9, 21.8. ESI-MS: 233 (M+H).

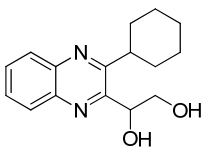


1-(3-tert-butylquinoxalin-2-yl)ethane-1,2-diol (7f): ^1H NMR (600 MHz, CDCl_3) δ ppm 8.06 (1H, d, J = 8.4 Hz), 7.99 (1H, d, J = 7.8 Hz), 7.71-7.76 (2H, m), 5.42-5.44 (1H, m), 3.97 (1H, dd, J = 11.6, 2.9 Hz), 3.83 (1H, dd, J = 11.6, 6.3 Hz), 1.59 (9H, s). ^{13}C NMR (100 MHz, CDCl_3) δ ppm 161.1, 154.7, 141.1, 139.3, 130.2, 129.8, 129.5, 128.2, 71.6, 68.1, 39.1, 30.6. HRMS (ESI+): Found 247.1455 Calc'd 247.1447 (M+H).



1-(3-cyclopropylquinoxalin-2-yl)ethane-1,2-diol (7g): ^1H NMR (500 MHz, CDCl_3) δ ppm 8.01 (1H, dd, J = 8.0, 1.1 Hz), 7.96 (1H, dd, J = 8.3, 1.2 Hz), 7.65-7.72 (2H, m), 5.36-5.39 (1H, m),

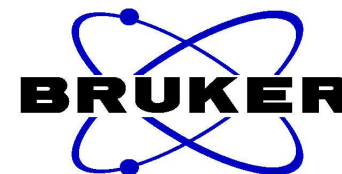
4.85 (1H, d, $J = 6.0$ Hz), 4.17 (1H, d, $J = 11.2$ Hz), 3.84 (1H, dd, $J = 6.0, 11.6$ Hz), 2.78 (1H, s, br), 2.27-2.32 (1H, m), 1.48-1.52 (1H, m), 1.23-1.27 (1H, m), 1.15-1.21 (2H, m). ^{13}C NMR (125 MHz, CDCl_3) δ ppm 156.2, 152.3, 142.3, 138.9, 129.9, 129.1, 128.8, 128.5, 71.1, 66.6, 13.8, 11.9, 10.6. HRMS (ESI⁺): Found 231.1133 Calc'd 231.1134 (M+H).



1-(3-cyclohexylquinoxalin-2-yl)ethane-1,2-diol (7h): ^1H NMR (500 MHz, CDCl_3) δ ppm 8.09 (1H, d, $J = 8.0$ Hz), 8.02 (1H, d, $J = 8.0$ Hz), 7.70-7.76 (2H, m), 5.21 (1H, s, br), 4.66 (1H, s, br), 4.01-4.04 (1H, m), 3.75 (1H, dd, $J = 11.5, 6.1$ Hz), 3.01-3.07 (1H, m), 1.76-1.98 (6H, m), 1.44-1.49 (4H, m). ^{13}C NMR (125 MHz, CDCl_3) δ ppm 159.6, 152.0, 142.5, 139.4, 130.0, 129.6, 129.1, 128.5, 70.7, 67.3, 41.7, 33.2, 32.0, 26.8, 26.1. HRMS (ESI⁺): Found 273.1618 Calc'd 273.1603 (M+H).

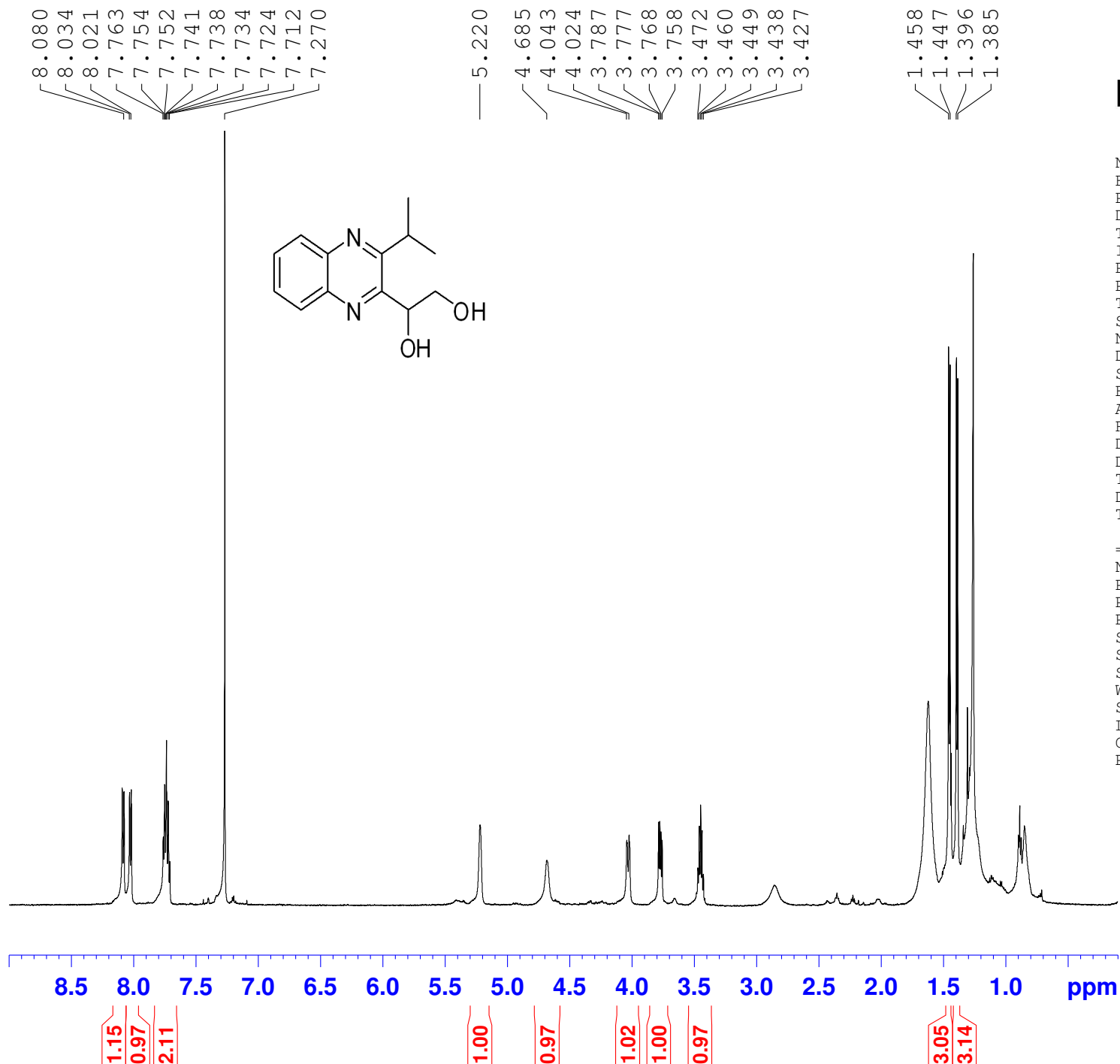
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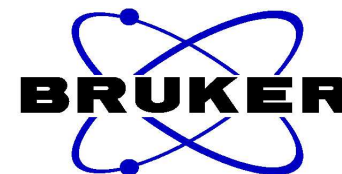
1. Jiang, N., Wang, J. (2002). DBU-promoted condensation of acyldiazomethanes to aldehydes and imines under catalytic conditions. *Tet. Lett.* 54 1285-1287



NAME i-Pr_diamine_purif
EXPNO 1
PROCNO 1
Date_ 20090724
Time 16.45
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 48074
SOLVENT CDC13
NS 32
DS 2
SWH 7812.500 Hz
FIDRES 0.162510 Hz
AQ 3.0767860 sec
RG 645
DW 64.000 usec
DE 6.50 usec
TE 295.1 K
D1 3.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.00 usec
PL1 -1.00 dB
PL1W 20.68129539 W
SFO1 600.1336008 MHz
SI 65536
SF 600.1300056 MHz
WDW EM
SSB 0
LB 0.60 Hz
GB 0
PC 3.00

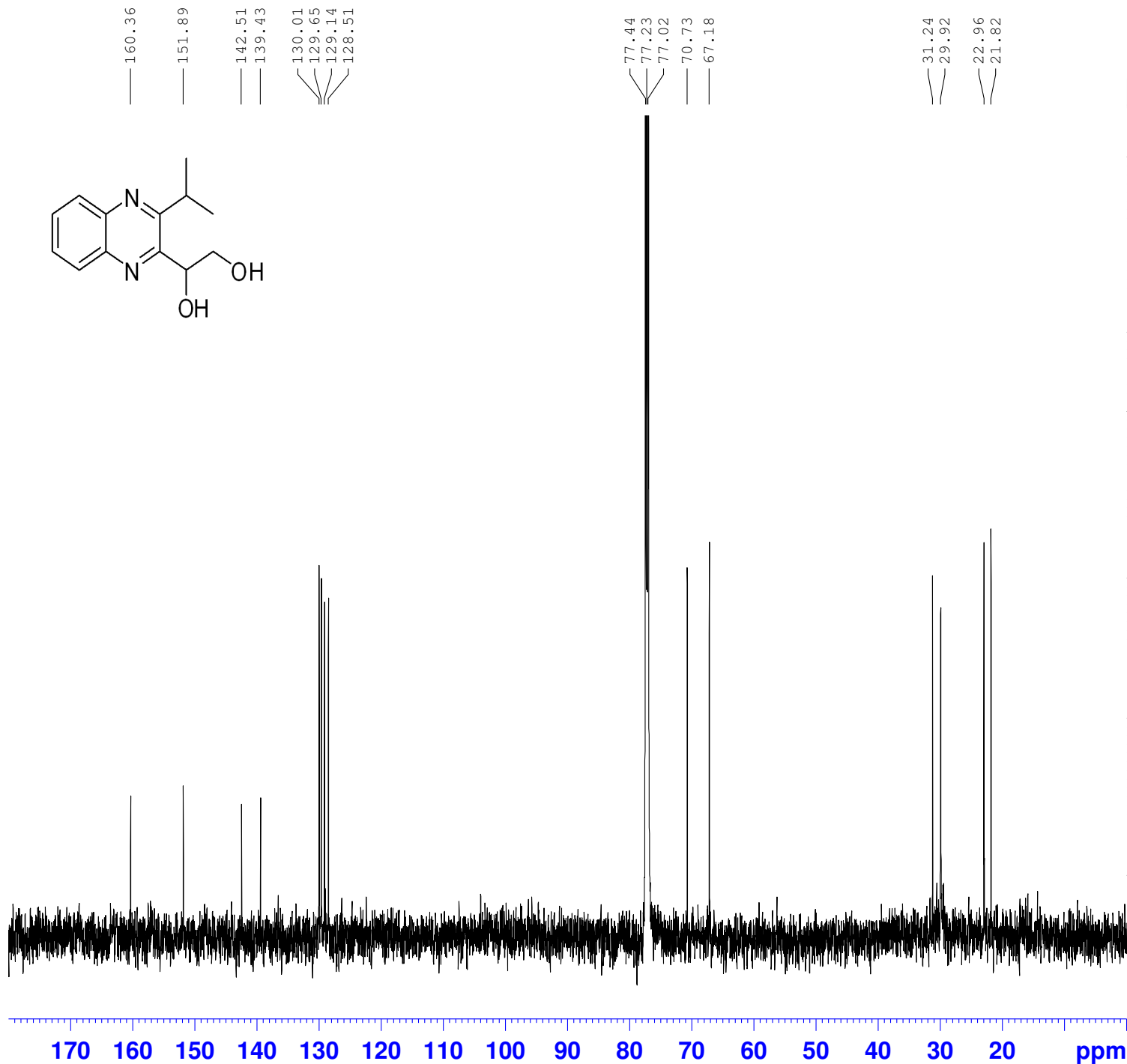
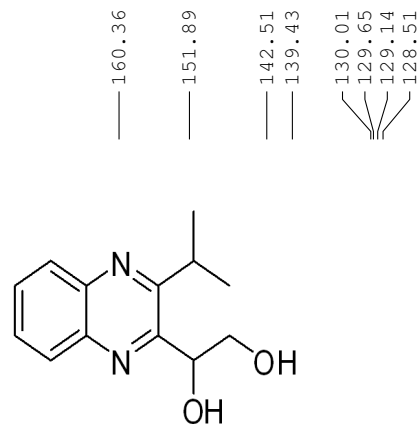




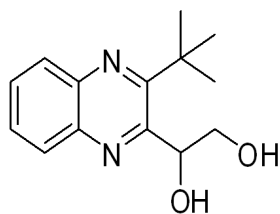
NAME i-Pr_diamine_purif
EXPNO 2
PROCNO 1
Date_ 20090724
Time 18.01
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgdc30
TD 66560
SOLVENT CDC13
NS 4670
DS 8
SWH 28846.154 Hz
FIDRES 0.433386 Hz
AQ 1.1537566 sec
RG 2050
DW 17.333 usec
DE 6.50 usec
TE 301.1 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 14

===== CHANNEL f1 =====
NUC1 13C
P1 7.60 usec
PL1 -1.00 dB
PL1W 92.65473175 W
SFO1 150.9163903 MHz

===== CHANNEL f2 =====
CPDPRG2 garp
NUC2 1H
PCPD2 66.00 usec
PL2 -1.00 dB
PL12 11.99 dB
PL2W 20.68129539 W
PL12W 1.03890967 W
SFO2 600.1327006 MHz
SI 65536
SF 150.9027740 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

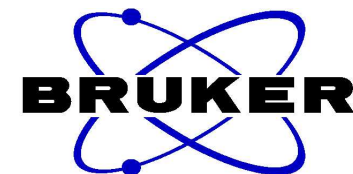


161.07
154.72
141.09
139.25
130.15
129.81
129.48
128.15



77.44
77.23
77.02
71.55
68.06

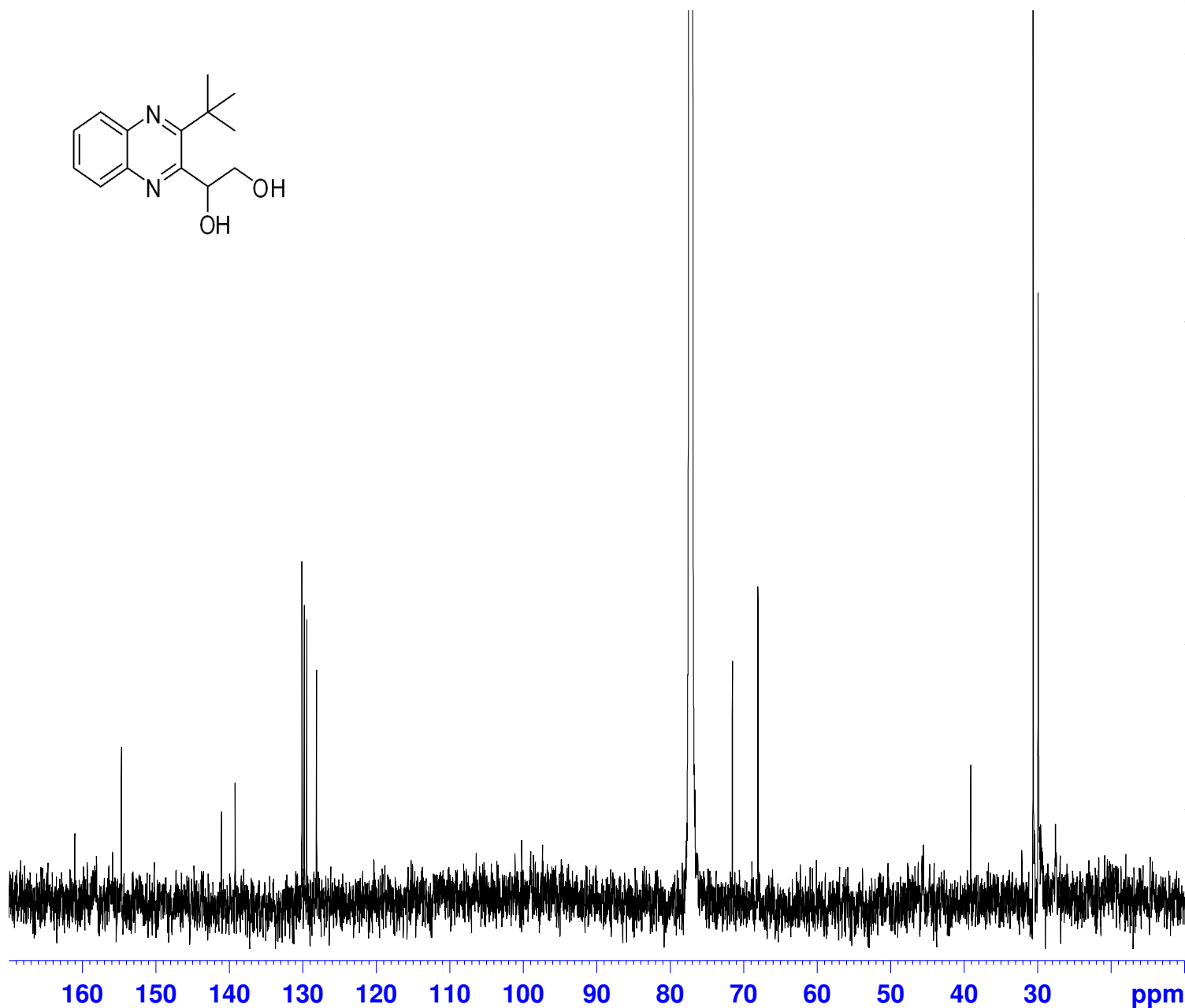
39.10
30.61
29.93

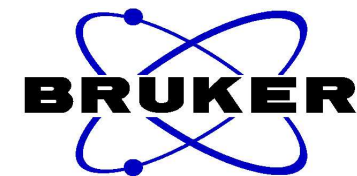
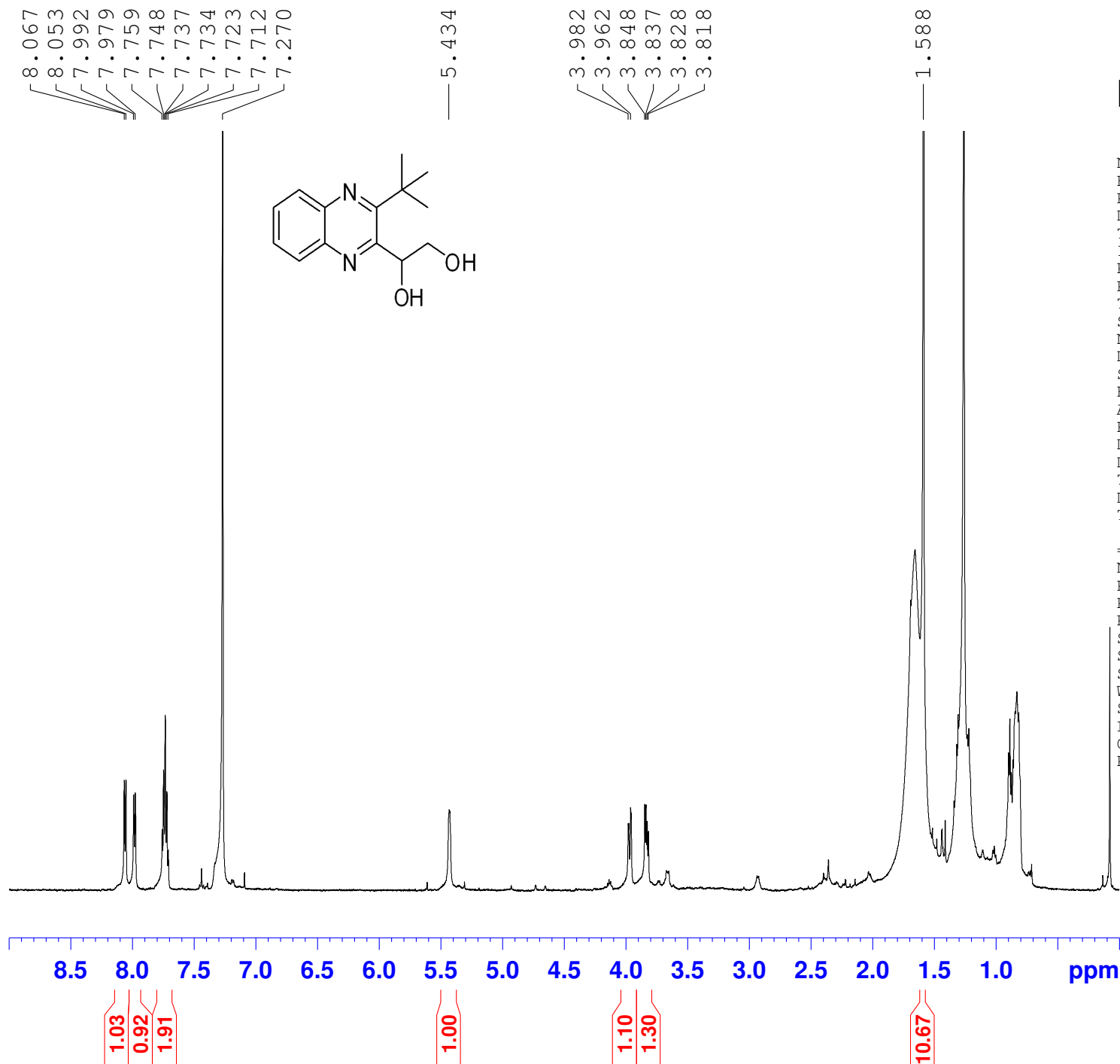


NAME t-Bu_diamine_purif
EXPNO 2
PROCNO 1
Date_ 20090723
Time 10.15
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgdc30
TD 66560
SOLVENT CDCl3
NS 40610
DS 8
SWH 32051.281 Hz
FIDRES 0.481540 Hz
AQ 1.0383860 sec
RG 16400
DW 15.600 usec
DE 6.50 usec
TE 298.8 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 11

===== CHANNEL f1 =====
NUC1 13C
P1 7.60 usec
PL1 -1.00 dB
PL1W 92.65473175 W
SFO1 150.9178993 MHz

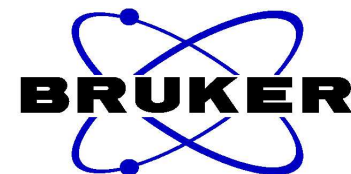
===== CHANNEL f2 =====
CPDPRG2 garp
NUC2 1H
PCPD2 66.00 usec
PL2 -1.00 dB
PL12 11.99 dB
PL2W 20.68129539 W
PL12W 1.03890967 W
SFO2 600.1327006 MHz
SI 65536
SF 150.9027735 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40





NAME t-Bu_diamine_purif
EXPNO 1
PROCNO 1
Date_ 20090723
Time 9.52
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 48074
SOLVENT CDCl3
NS 128
DS 2
SWH 7812.500 Hz
FIDRES 0.162510 Hz
AQ 3.0767860 sec
RG 114
DW 64.000 usec
DE 6.50 usec
TE 294.4 K
D1 4.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.00 usec
PL1 -1.00 dB
PL1W 20.68129539 W
SFO1 600.1336008 MHz
SI 65536
SF 600.1300056 MHz
WDW EM
SSB 0
LB 0.60 Hz
GB 0
PC 3.00

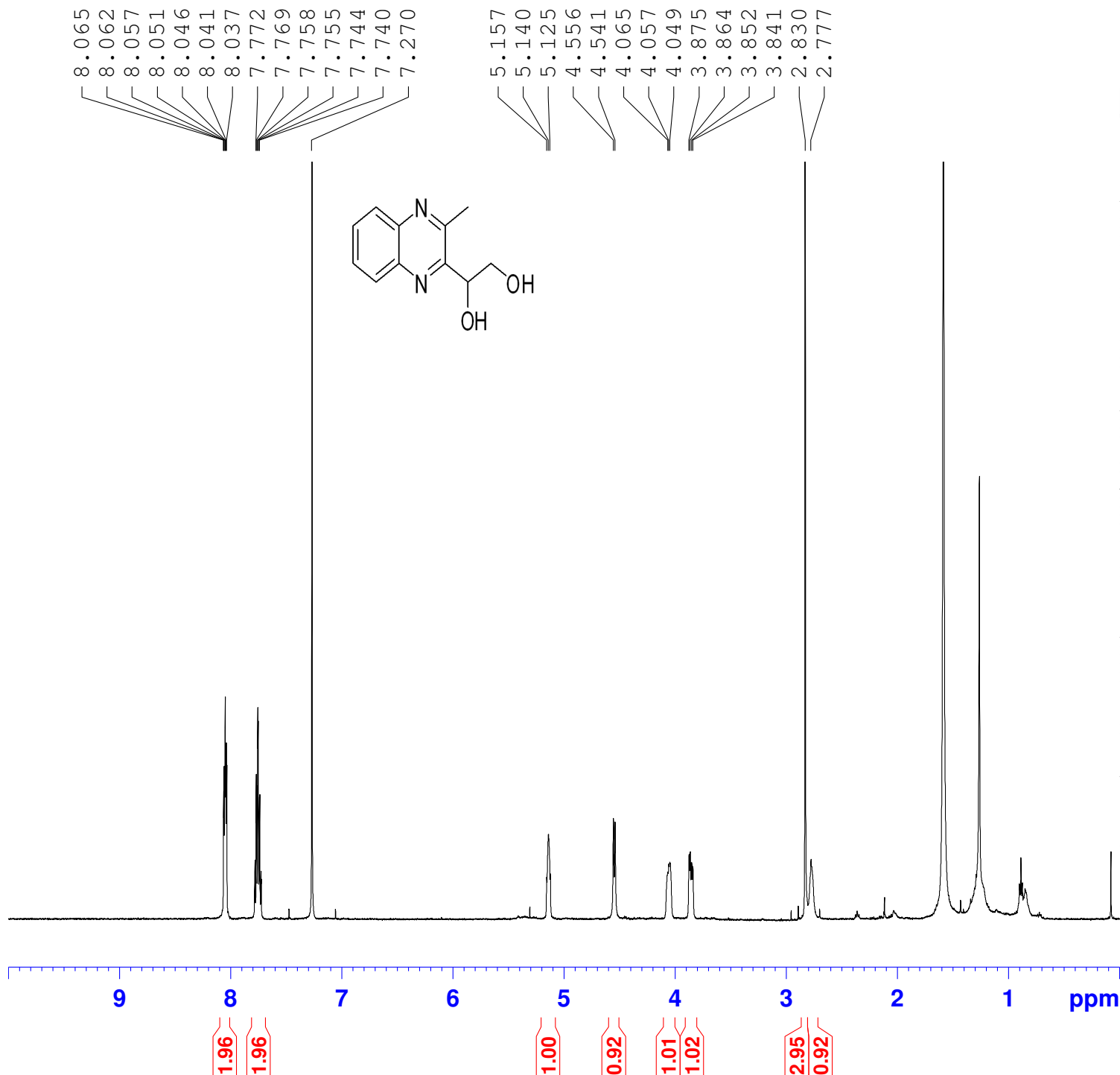


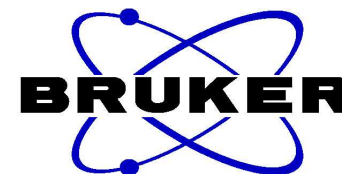
Current Data Parameters
NAME JS_AI2_diamine
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090716
Time 20.07
INSTRUM spect
PROBHD 5 mm BBO2 new
PULPROG zg
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 6510.417 Hz
FIDRES 0.099341 Hz
AQ 5.0332146 sec
RG 181
DW 76.800 usec
DE 9.00 usec
TE 296.1 K
D1 4.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 11.10 usec
PL1 0.00 dB
SFO1 500.1330008 MHz

F2 - Processing parameters
SI 32768
SF 500.1300082 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00





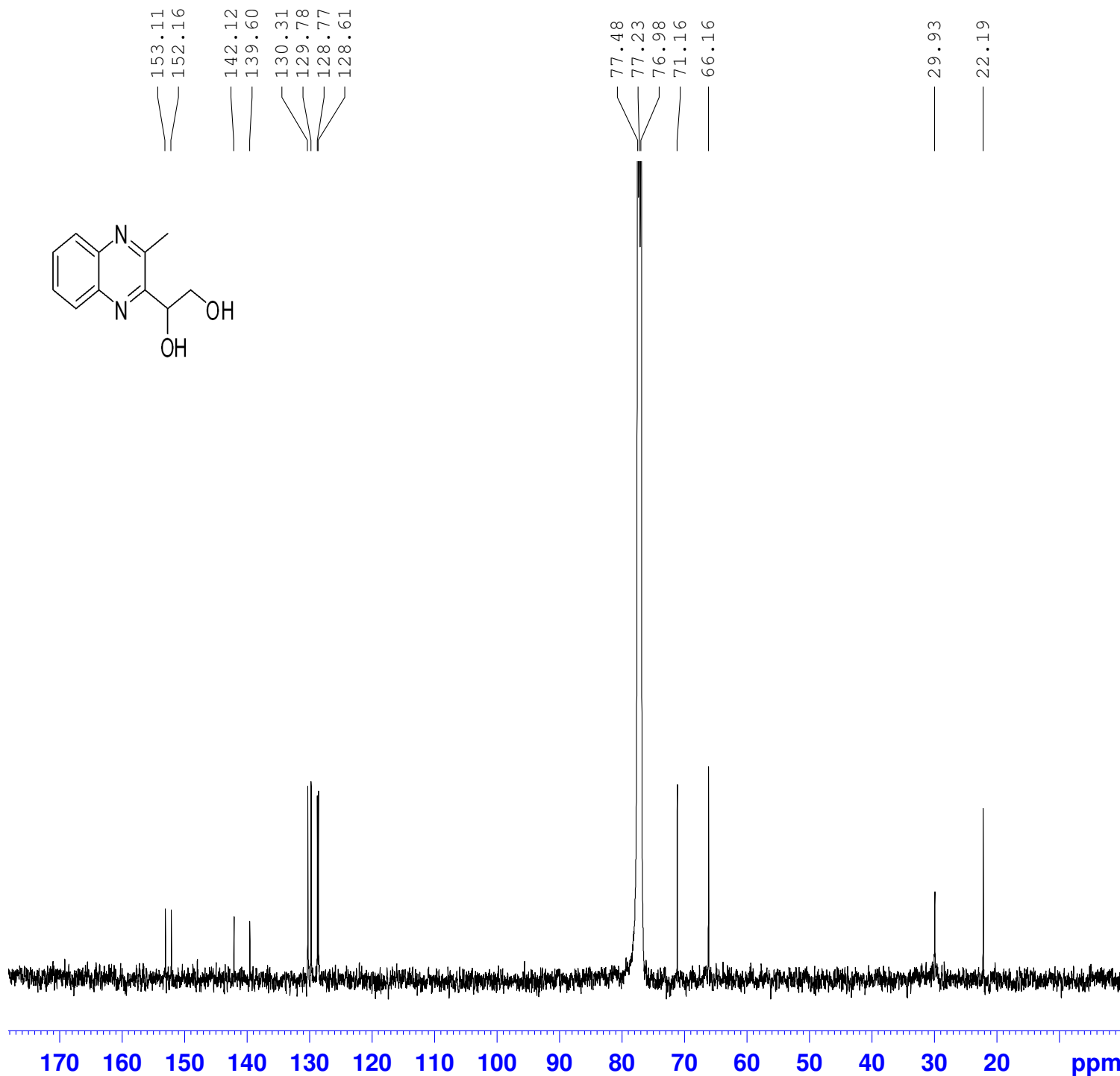
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NAME JS_AI2_diamine
EXPNO 2
PROCNO 1

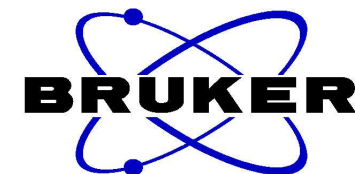
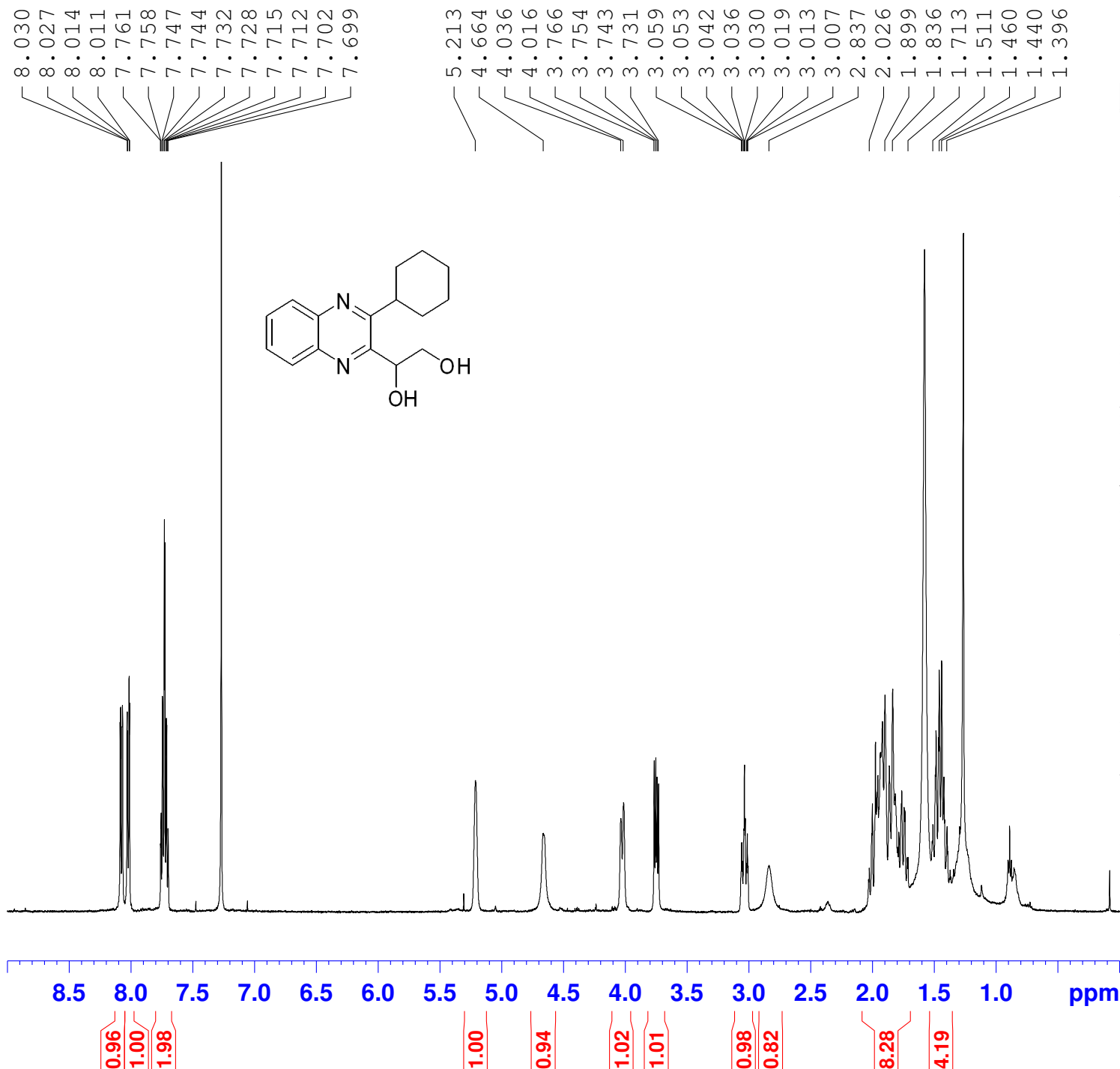
F2 - Acquisition Parameters
Date_ 20090716
Time 20.59
INSTRUM spect
PROBHD 5 mm BBO2 new
PULPROG zgdc30
TD 94336
SOLVENT CD2Cl2
NS 18260
DS 4
SWH 30120.482 Hz
FIDRES 0.319289 Hz
AQ 1.5660276 sec
RG 5160.6
DW 16.600 usec
DE 6.00 usec
TE 299.8 K
D1 1.00000000 sec
d11 0.03000000 sec
TD0 9

===== CHANNEL f1 =====
NUC1 13C
P1 13.10 usec
PL1 3.00 dB
SFO1 125.7722511 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 60.00 usec
PL2 0.00 dB
PL12 17.80 dB
SFO2 500.1325007 MHz

F2 - Processing parameters
SI 65536
SF 125.7577601 MHz
WDW EM
SSB 0
LB 4.00 Hz
GB 0
PC 1.40





Current Data Parameters
NAME JS_chexane_diamine
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090715
Time 23.57
INSTRUM spect
PROBHD 5 mm BBO2 new
PULPROG zg
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 6510.417 Hz
FIDRES 0.099341 Hz
AQ 5.0332146 sec
RG 256
DW 76.800 usec
DE 9.00 usec
TE 300.0 K
D1 4.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 11.10 usec
PL1 0.00 dB
SFO1 500.1330008 MHz

F2 - Processing parameters
SI 32768
SF 500.1300080 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



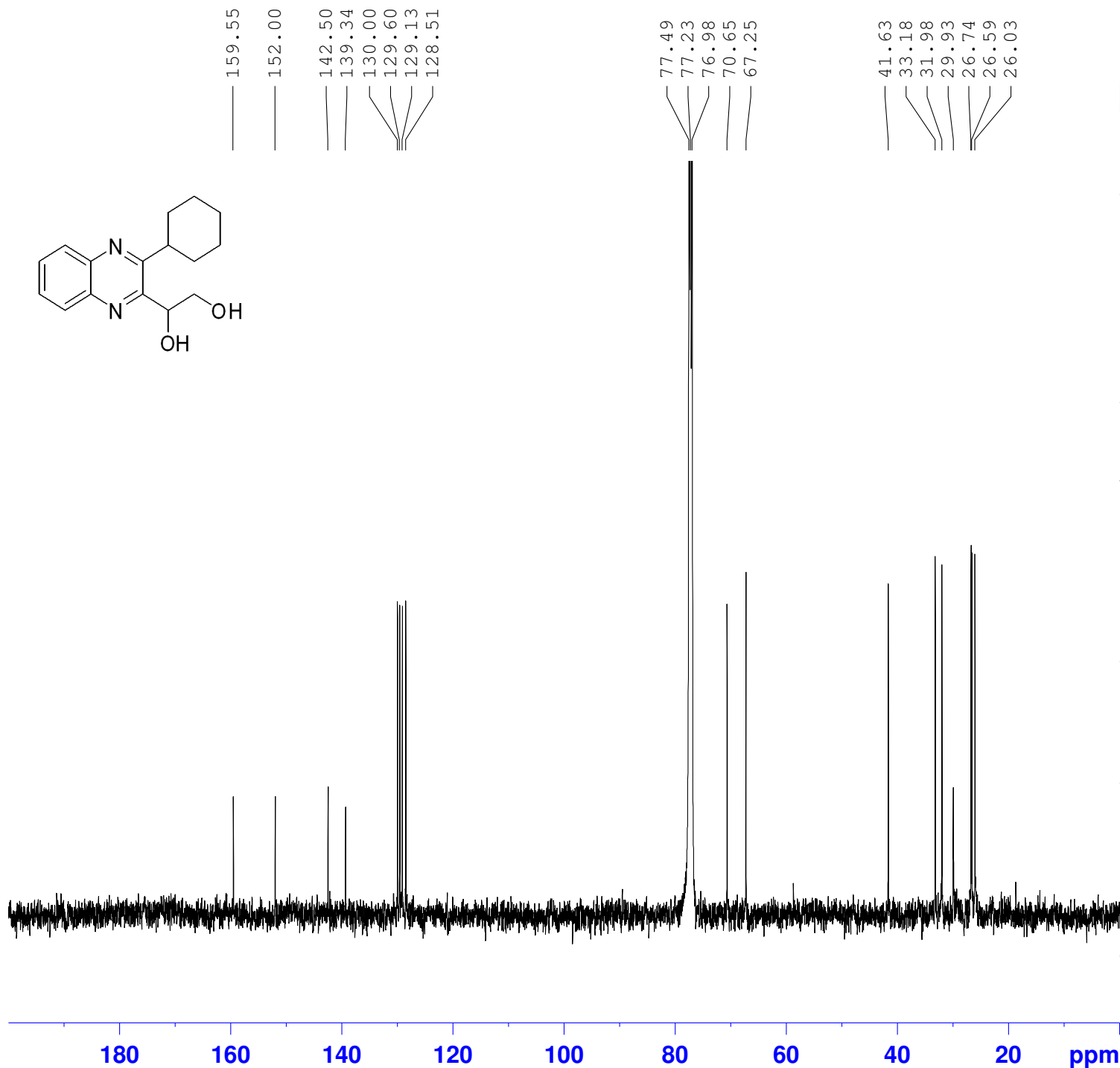
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NAME JS_chexane_diamine
EXPNO 2
PROCNO 1

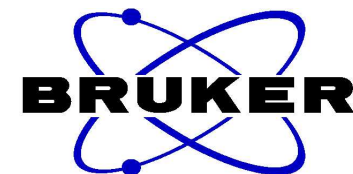
F2 - Acquisition Parameters
Date_ 20090716
Time 0.13
INSTRUM spect
PROBHD 5 mm BBO2 new
PULPROG zgpg30
TD 65536
SOLVENT C6D6
NS 15360
DS 32
SWH 30120.482 Hz
FIDRES 0.459602 Hz
AQ 1.0879476 sec
RG 10321.3
DW 16.600 usec
DE 6.50 usec
TE 301.9 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 15

===== CHANNEL f1 =====
NUC1 13C
P1 13.10 usec
PL1 3.00 dB
SFO1 125.7722011 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 60.00 usec
PL2 0.00 dB
PL12 17.80 dB
PL13 20.00 dB
SFO2 500.1325007 MHz

F2 - Processing parameters
SI 32768
SF 125.7577603 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40



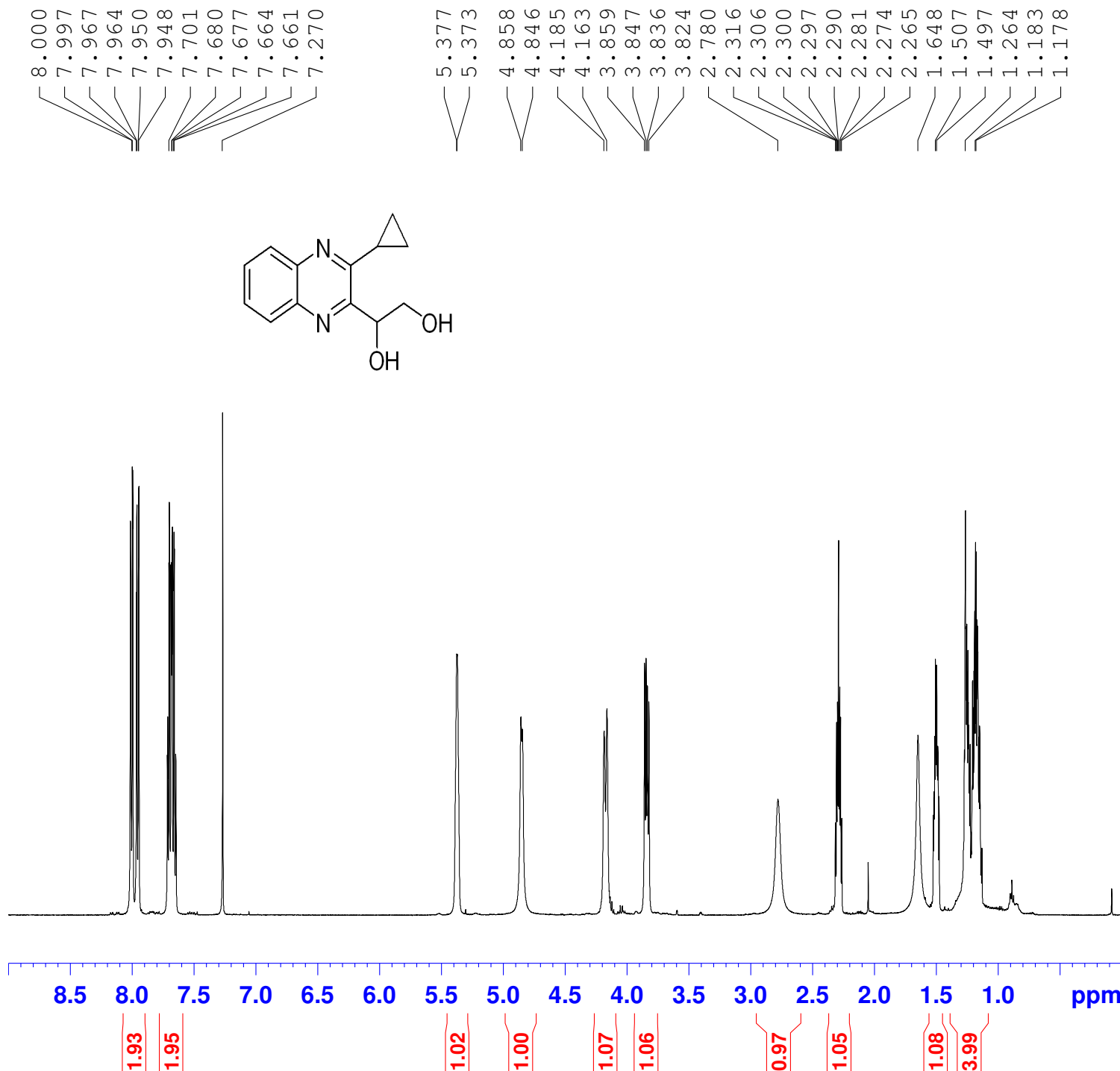


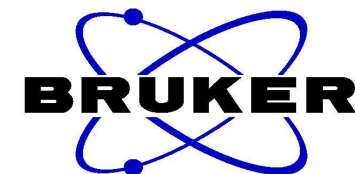
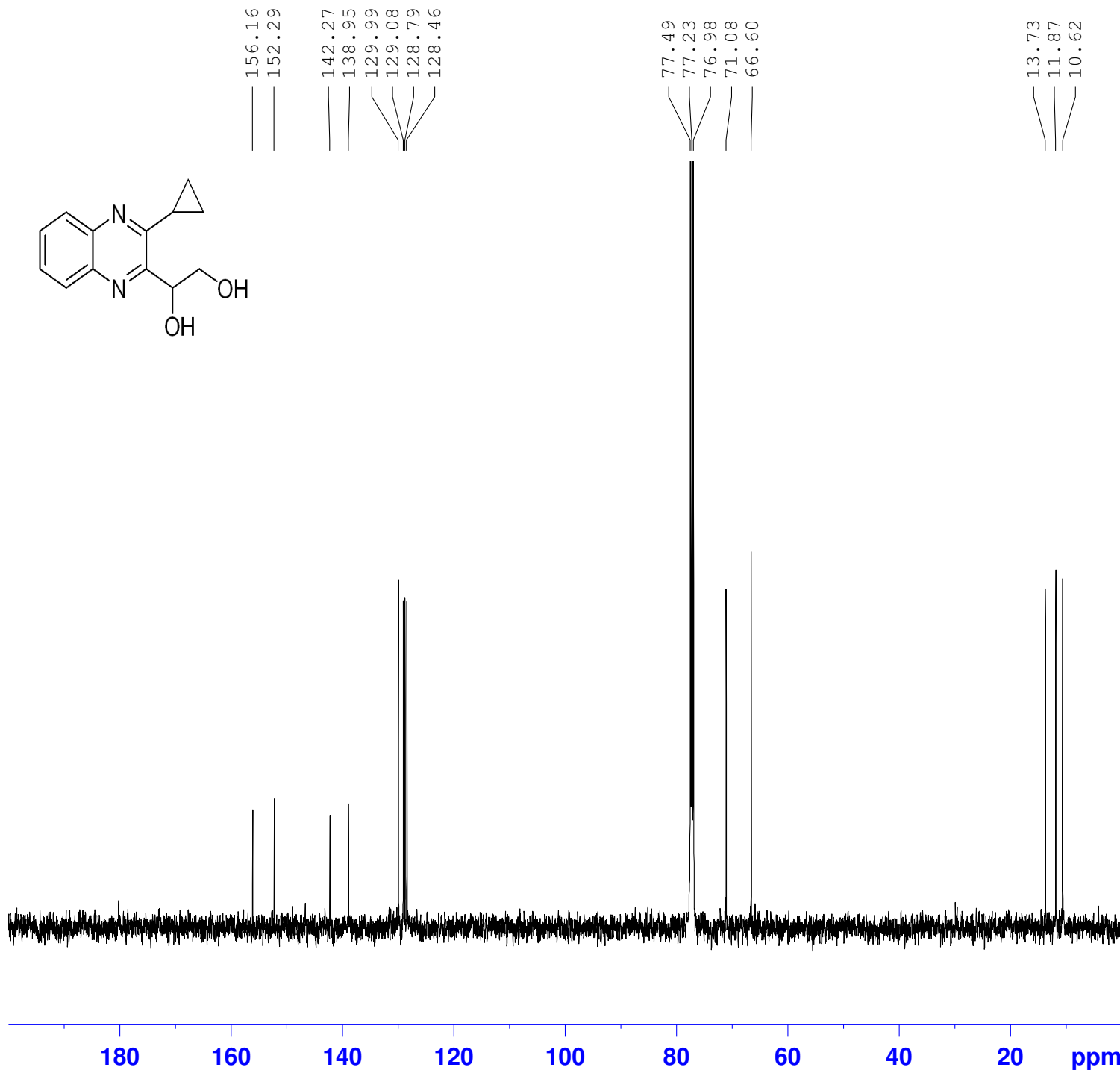
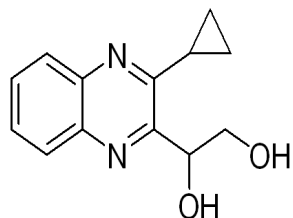
Current Data Parameters
NAME JS_cpropane_diamine
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090715
Time 23.25
INSTRUM spect
PROBHD 5 mm BBO2 new
PULPROG zg
TD 65536
SOLVENT CDC13
NS 14
DS 2
SWH 6510.417 Hz
FIDRES 0.099341 Hz
AQ 5.0332146 sec
RG 181
DW 76.800 usec
DE 9.00 usec
TE 301.4 K
D1 4.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 11.10 usec
PL1 0.00 dB
SFO1 500.1330008 MHz

F2 - Processing parameters
SI 32768
SF 500.1300082 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00





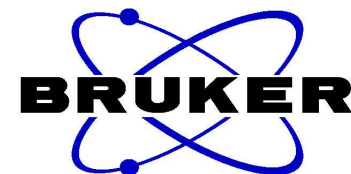
Current Data Parameters
NAME JS_cpropane_diamine
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090715
Time 23.32
INSTRUM spect
PROBHD 5 mm BBO2 new
PULPROG zgpg30
TD 65536
SOLVENT C6D6
NS 580
DS 32
SWH 30120.482 Hz
FIDRES 0.459602 Hz
AQ 1.0879476 sec
RG 9195.2
DW 16.600 usec
DE 6.50 usec
TE 303.3 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 13.10 usec
PL1 3.00 dB
SFO1 125.7722011 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 60.00 usec
PL2 0.00 dB
PL12 17.80 dB
PL13 20.00 dB
SFO2 500.1325007 MHz

F2 - Processing parameters
SI 32768
SF 125.7577609 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

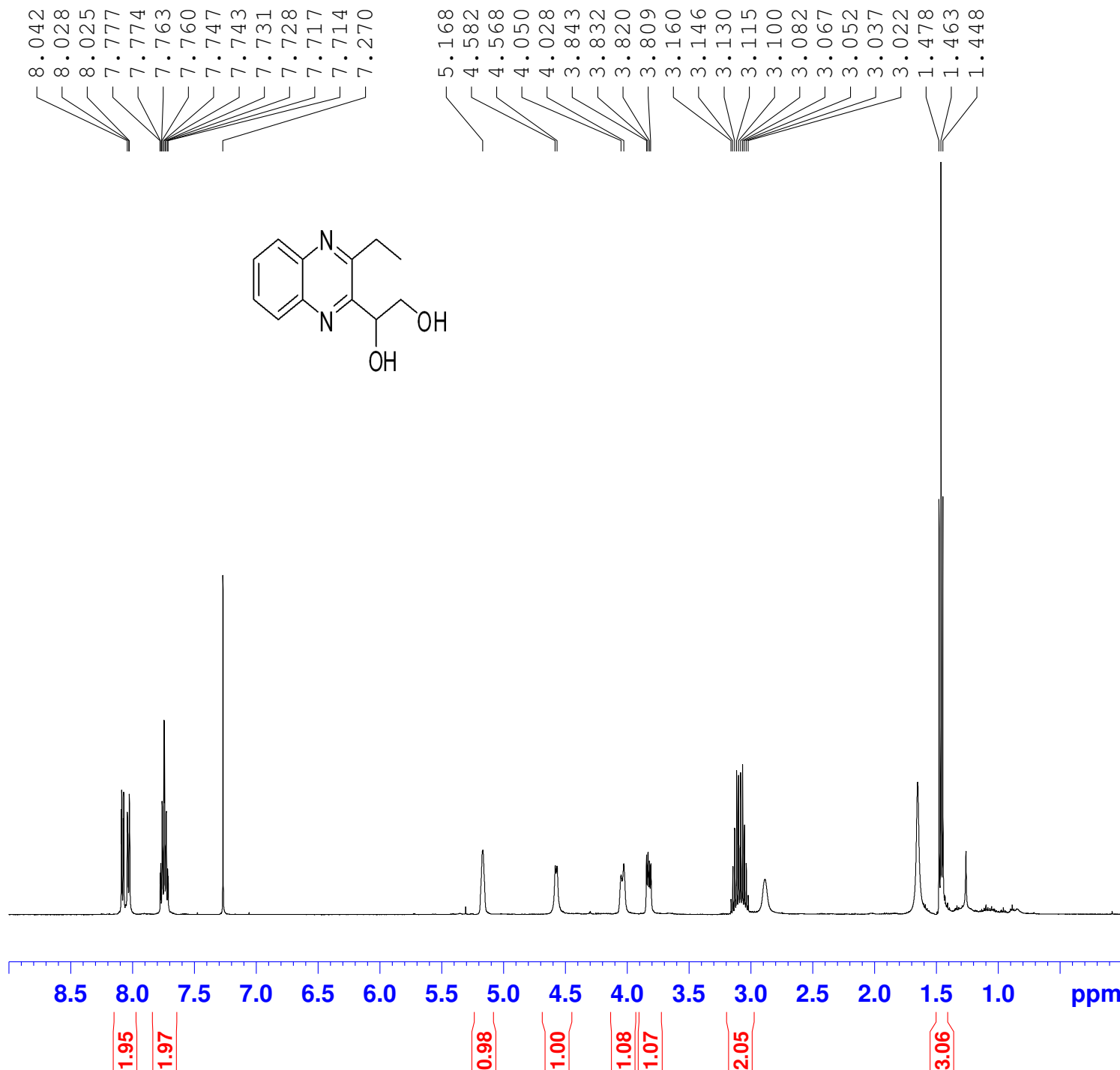
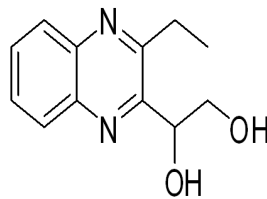


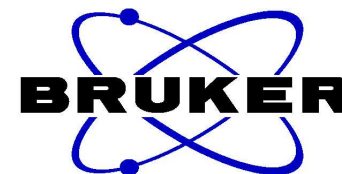
Current Data Parameters
NAME JS_Et_diamine
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090720
Time 21.28
INSTRUM spect
PROBHD 5 mm BBO2 new
PULPROG zg
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 6510.417 Hz
FIDRES 0.099341 Hz
AQ 5.0332146 sec
RG 181
DW 76.800 usec
DE 9.00 usec
TE 296.7 K
D1 4.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 11.10 usec
PL1 0.00 dB
SFO1 500.1330008 MHz

F2 - Processing parameters
SI 32768
SF 500.1300082 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00





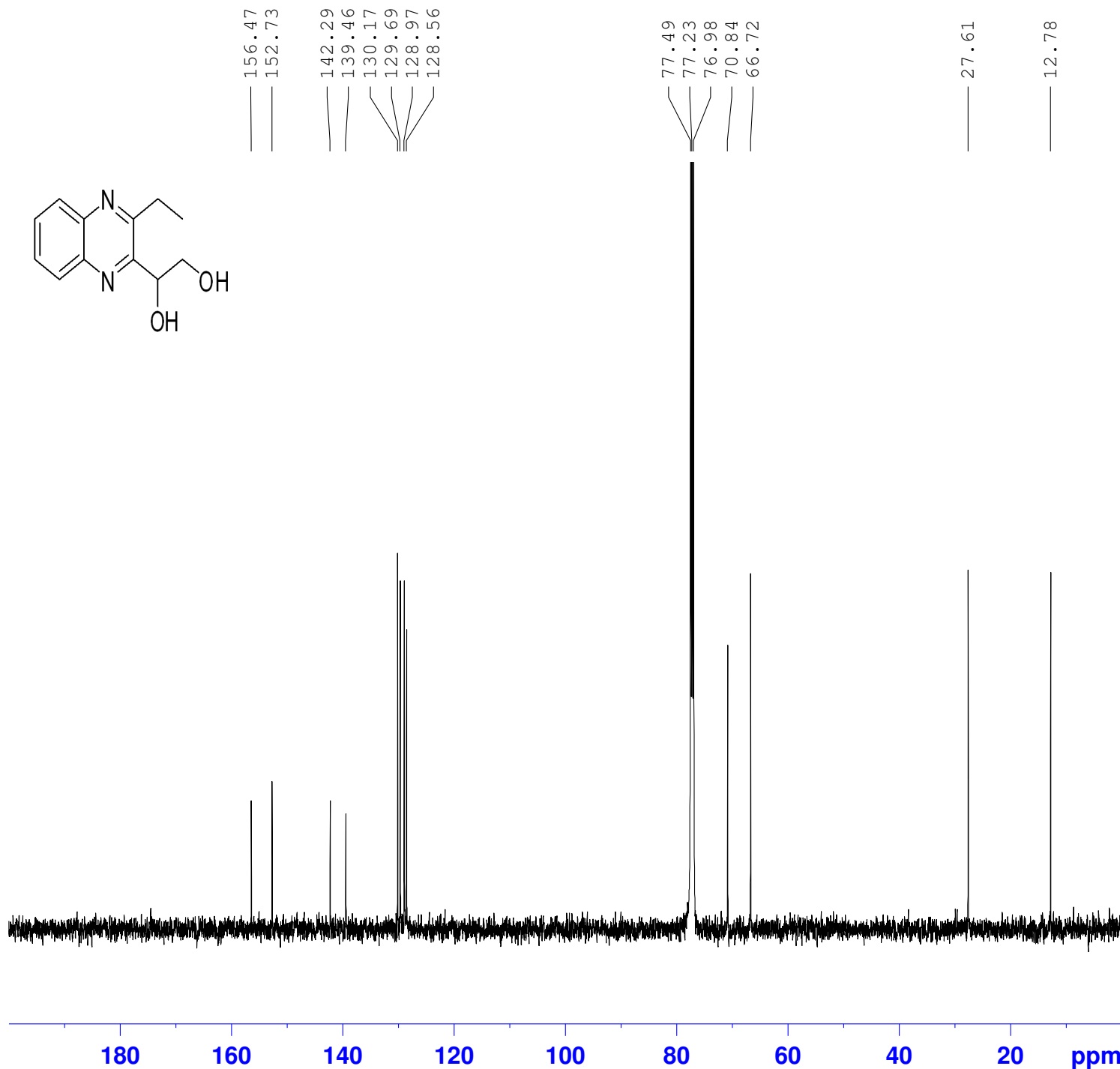
Current Data Parameters
NAME JS_Et_diamine
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090720
Time 22.54
INSTRUM spect
PROBHD 5 mm BBO2 new
PULPROG zgpg30
TD 65536
SOLVENT C6D6
NS 2362
DS 8
SWH 30120.482 Hz
FIDRES 0.459602 Hz
AQ 1.0879476 sec
RG 9195.2
DW 16.600 usec
DE 6.50 usec
TE 299.4 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 13.10 usec
PL1 3.00 dB
SFO1 125.7722011 MHz

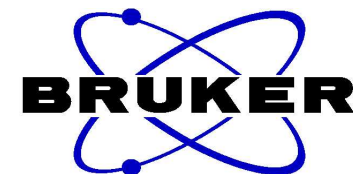
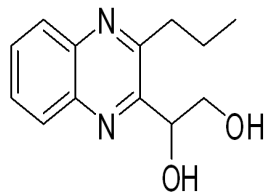
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 60.00 usec
PL2 0.00 dB
PL12 17.80 dB
PL13 20.00 dB
SFO2 500.1325007 MHz

F2 - Processing parameters
SI 32768
SF 125.7577618 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40



8.034
8.031
8.018
8.015
7.768
7.765
7.754
7.751
7.738
7.734
7.722
7.719
7.708
7.270

5.177
5.172
4.610
4.596
4.047
4.027
3.829
3.817
3.805
3.794
3.044
3.031
3.026
3.012
3.007
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1.088
1.073

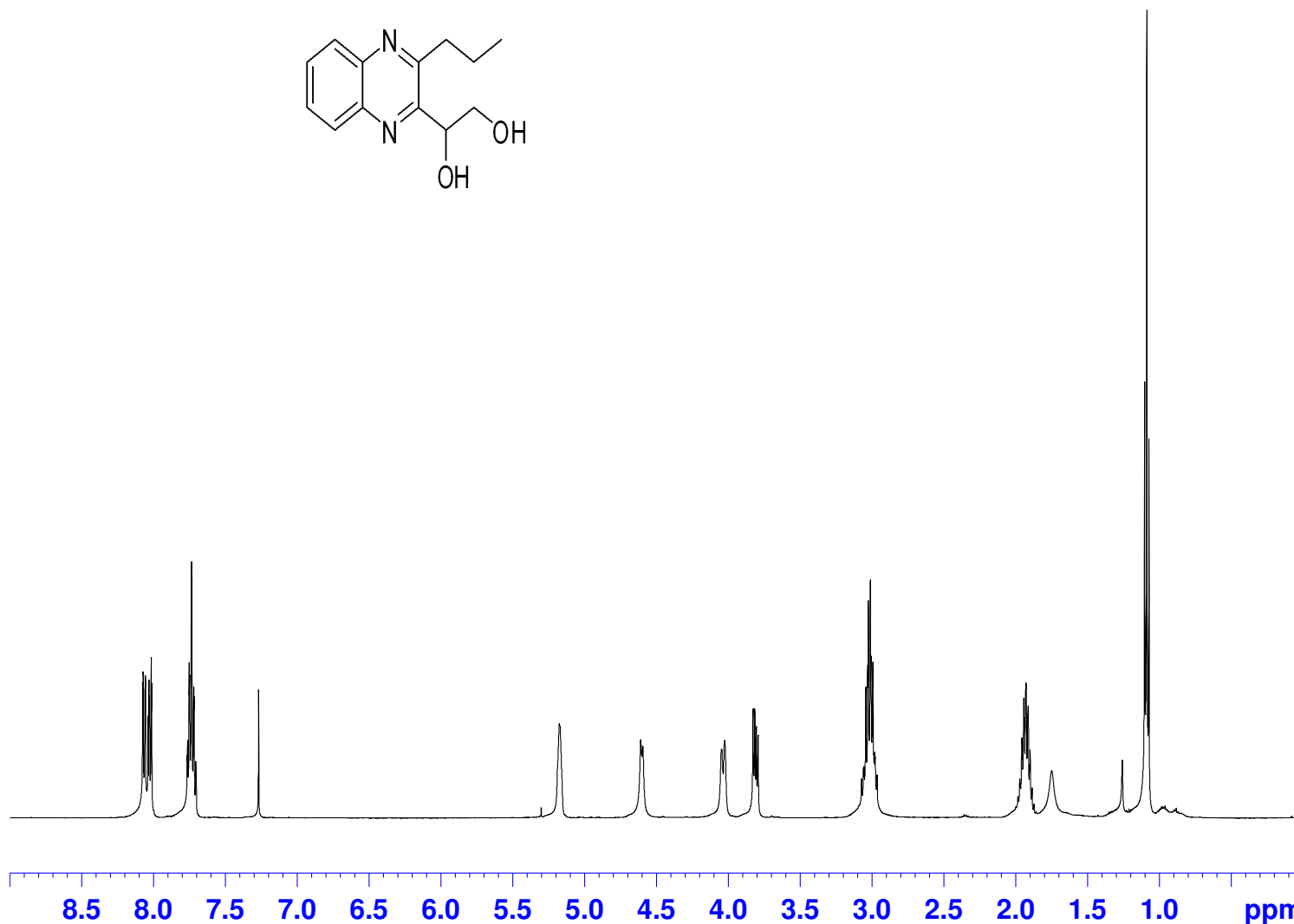


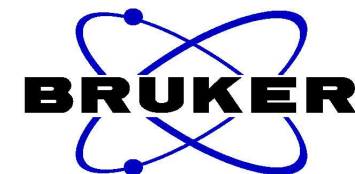
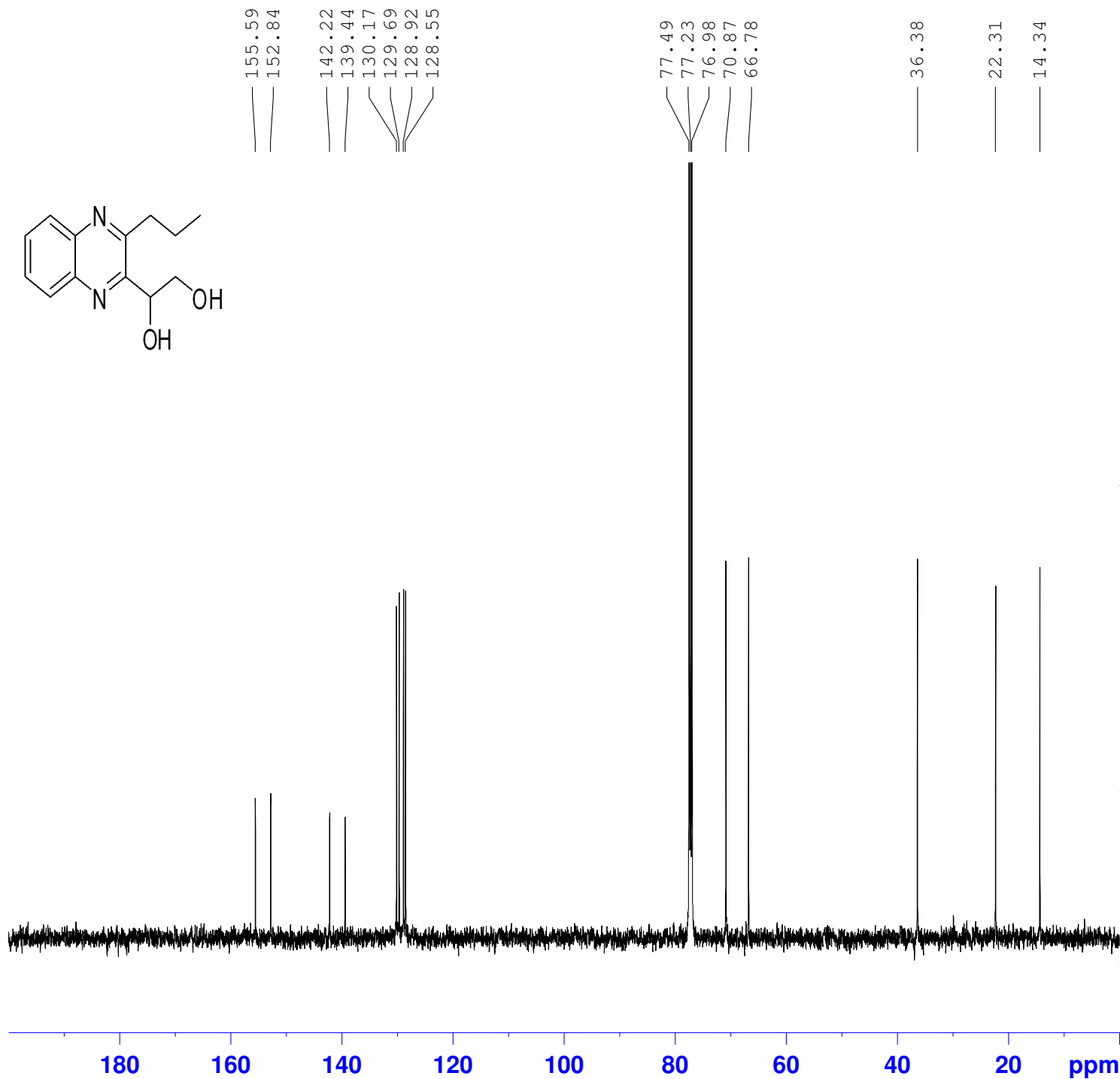
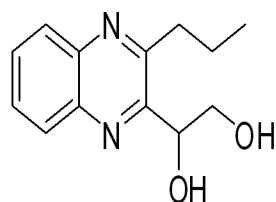
Current Data Parameters
NAME JS_i-pr_diamine
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090718
Time 13.08
INSTRUM spect
PROBHD 5 mm BBO2 new
PULPROG zg
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6510.417 Hz
FIDRES 0.099341 Hz
AQ 5.0332146 sec
RG 101.6
DW 76.800 usec
DE 9.00 usec
TE 296.1 K
D1 4.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 11.10 usec
PL1 0.00 dB
SFO1 500.1330008 MHz

F2 - Processing parameters
SI 32768
SF 500.1300079 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00





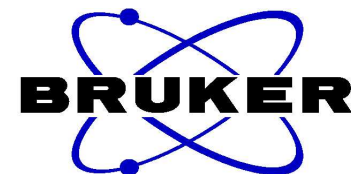
Current Data Parameters
NAME JS_i-pr_diamine
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090718
Time 13.47
INSTRUM spect
PROBHD 5 mm BBO2 new
PULPROG zgpg30
TD 65536
SOLVENT C6D6
NS 1024
DS 8
SWH 30120.482 Hz
FIDRES 0.459602 Hz
AQ 1.0879476 sec
RG 9195.2
DW 16.600 usec
DE 6.50 usec
TE 299.0 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 13.10 usec
PL1 3.00 dB
SFO1 125.7722011 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 60.00 usec
PL2 0.00 dB
PL12 17.80 dB
PL13 20.00 dB
SFO2 500.1325007 MHz

F2 - Processing parameters
SI 32768
SF 125.7577636 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

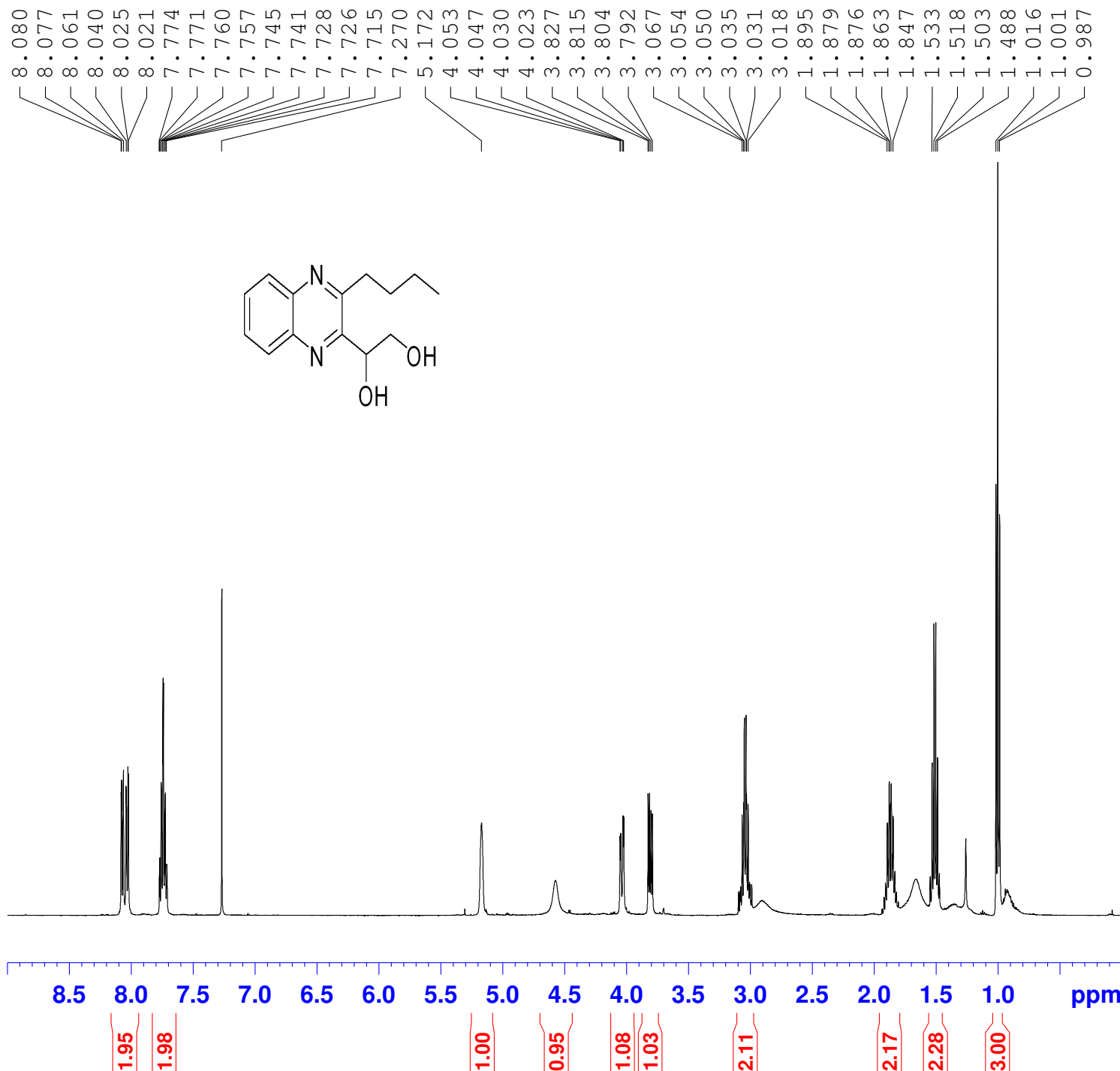


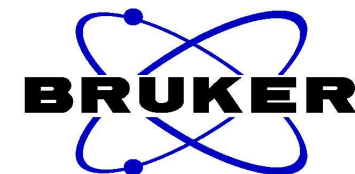
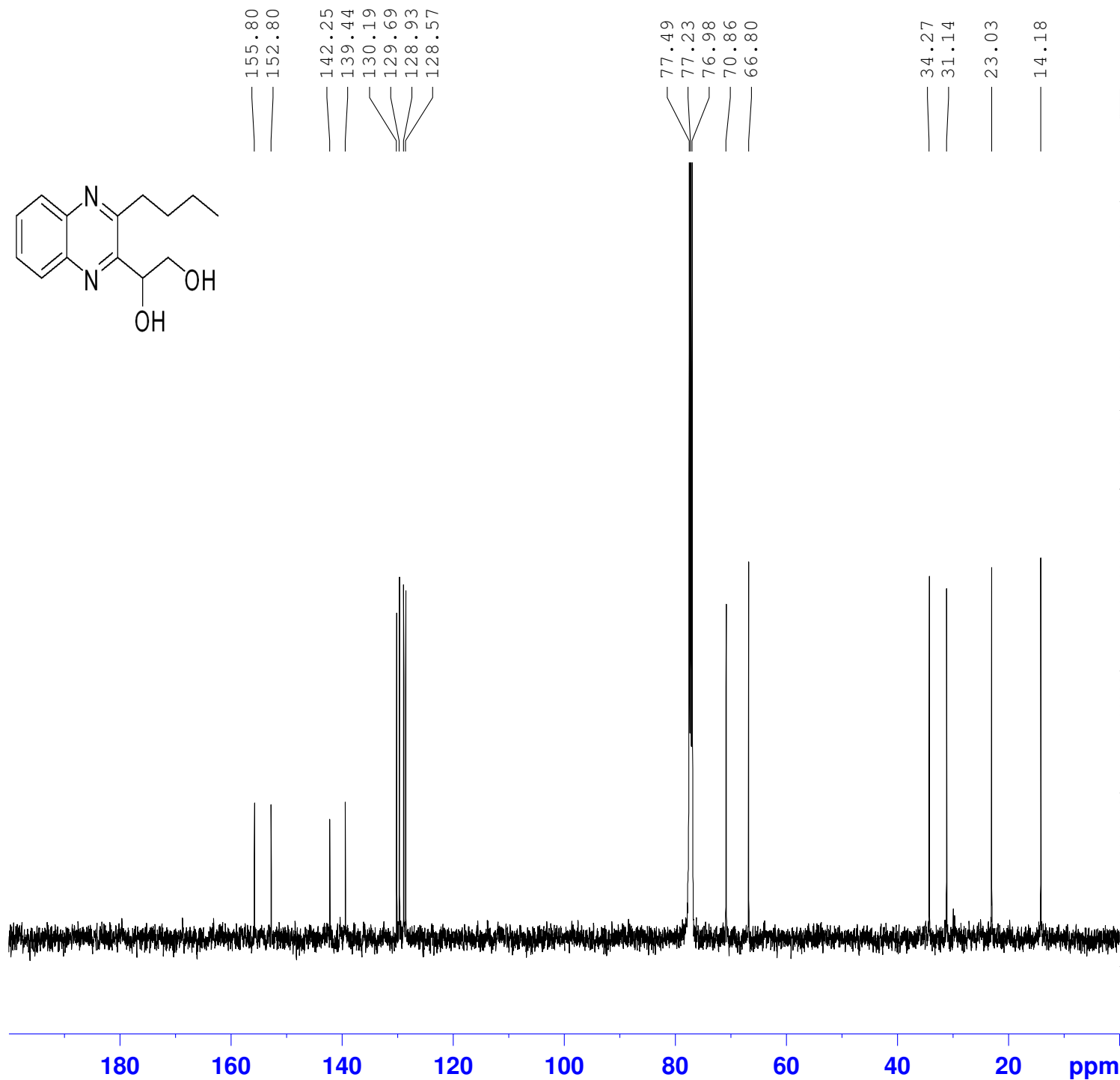
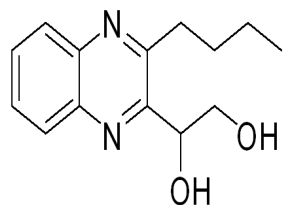
Current Data Parameters
NAME JS_n-Bu_diamine
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090718
Time 10.25
INSTRUM spect
PROBHD 5 mm BBO2 new
PULPROG zg
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 6510.417 Hz
FIDRES 0.099341 Hz
AQ 5.0332146 sec
RG 181
DW 76.800 usec
DE 9.00 usec
TE 296.0 K
D1 4.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 11.10 usec
PL1 0.00 dB
SFO1 500.1330008 MHz

F2 - Processing parameters
SI 32768
SF 500.1300081 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00





Current Data Parameters
NAME JS_n-Bu_diamine
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090718
Time 11.38
INSTRUM spect
PROBHD 5 mm BBO2 new
PULPROG zgpg30
TD 65536
SOLVENT C6D6
NS 1982
DS 8
SWH 30120.482 Hz
FIDRES 0.459602 Hz
AQ 1.0879476 sec
RG 9195.2
DW 16.600 usec
DE 6.50 usec
TE 299.1 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 13.10 usec
PL1 3.00 dB
SFO1 125.7722011 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 60.00 usec
PL2 0.00 dB
PL12 17.80 dB
PL13 20.00 dB
SFO2 500.1325007 MHz

F2 - Processing parameters
SI 32768
SF 125.7577619 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

