Supporting Information

Organocatalysis by *p*-sulfonic acid calix[4]arene: a convenient and efficient route to 2,3-dihydroquinazolin-4(1*H*)-ones in water

Matiur Rahman,[a] Irene ling,[a] Norbani Abdullah,[a] Rauzah Hashim*[a] and Alakananda Hajra*[b]

[a] Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia.

[b] Department of Chemistry, Visva-Bharati (A Central University), Santiniketan 731235, India.

E-mail: rauzah@um.edu.my or Email: alakananda.hajra@visva-bharati.ac.in

Table of Contents

EXPERIMENTAL

General: Melting points were determined on a glass disk with an electric hot plate and are uncorrected. ^{1}H NMR (400 MHz) and ^{13}C NMR (100 MHz) spectra were run in in $CDCl_3$ & DMSO-*d*6 solutions (Bruker Avance 400). Chemical shift were recorded as δ values in parts per million (ppm), and the signals were reported as s (singlet), d (doublet), t (triplet), m (multiplet) and coupling constants *J* were given in Hz. IR spectra were taken in a Perkin Elmer FTIR-Spectrum 400. Commercially available substrates were freshly distilled before the reaction. Solvents, reagents and chemicals were purchased from Sigma Aldrich and Merck.

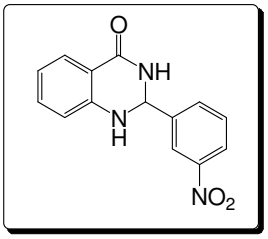
General procedure 2,3-dihydroquinazolin-4(1*H*)-one:

p-Sulfonic acid calix[4]arene (1 mol%) was added to a solution of 2-aminobenzamide (1 mmol) and aldehyde/ketone (1 mmol) in water (1 mL) and the mixture was stirred at room temperature for a specified period of time. After completion of the reaction, cold distilled water (5 mL) was added to the reaction mixture. Then the product was filtered off and the *p*-sulfonic acid calix[4]arene was recovered by evaporating the water. The recovered *p*-sulfonic acid calix[4]arene was reused for a subsequent fresh batch of the reaction after reactivation. The crude product was recrystallized from hot ethanol to afford the pure product.

Single crystal XRD data of 2-(3-nitrophenyl)-2,3-dihydroquinazolin-4(1*H*)-one (3n):

Single crystal suitable for X-ray diffraction of (**3n**) was grown from ethanol. The crystals were carefully chosen using a stereo zoom microscope supported by a rotatable polarizing stage. The data was collected at room temperature on Bruker- AXS SMART APEX II, CCD detector.

Crystal data of 3n:



$C_{14}H_{11}N_3O_3$, M = 269.26, colorless plate, 0.30 × 0.25 × 0.20 mm^3, monoclinic, space group $P2_1/n$ (No. 14), a = 10.9782(11), b = 9.8343(8), c = 11.7533(11) Å, α = 90, β = 109.846(10), γ = 90°, V = 1193.6(2) $Å^3$, Z = 4, D_c = 1.498 g/cm^3, F_{000} = 560, MoKα radiation, λ = 0.71073 Å, T = 100(2)K, $2\theta_{max}$ = 60.2°, 7235 reflections collected, 3141 unique (R_{int} = 0.0326). Final *GooF* = 1.002, *R1* = 0.0522, *wR2* = 0.1342, *R* indices based on 3141 reflections with with I > 2σ(I) (refinement on F^2), 181 parameters, 0 restraints. Lp and absorption corrections applied. Crystallographic data for **3n** have been deposited with the Cambridge Crystallographic Data Center as supplementary publication number CCDC 1026285.

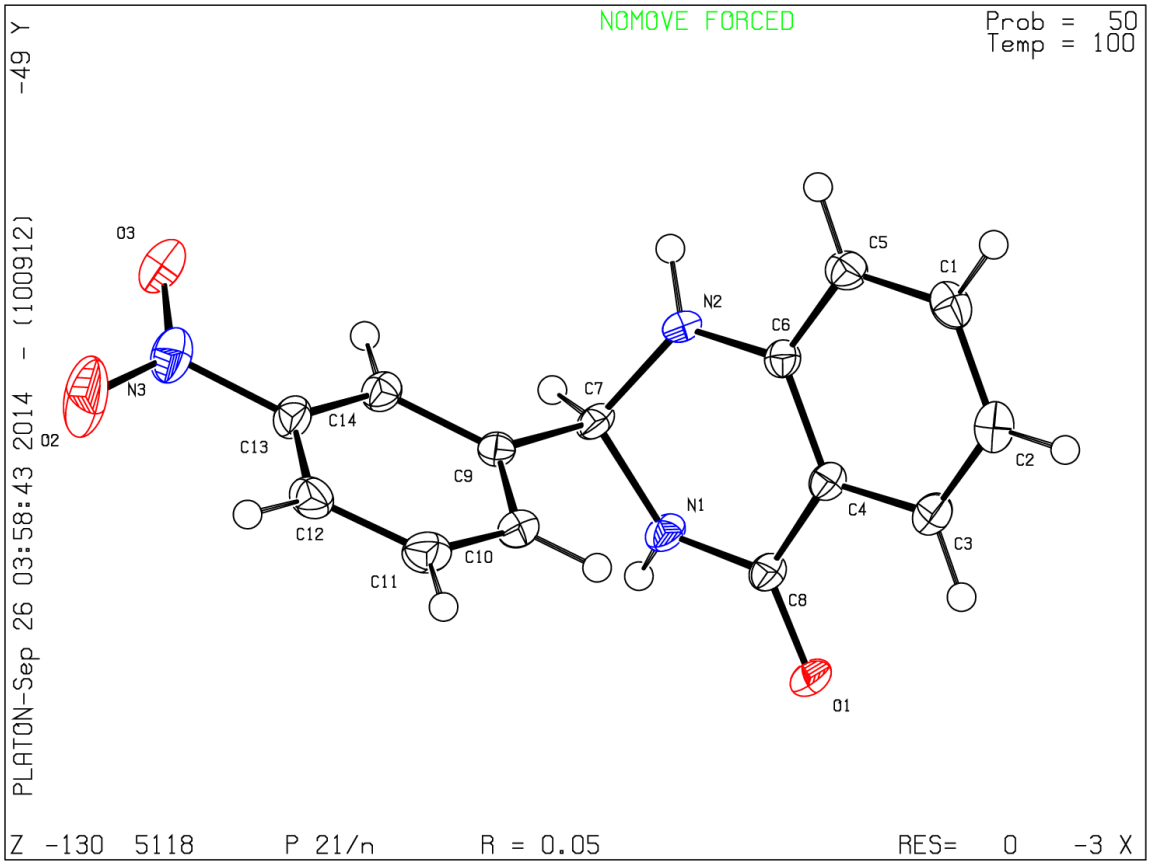


Figure 1: ORTEP view of the compound **3n.**

E-factor Calculations:[1]

Table 1 Synthesis of 2,3-dihydroquinazolin-4(1*H*)-ones.: E-factor value for this protocol.[a]

Entry	Anthranilamide (mmol)	Aldehyde (mmol)	Product	Isolated yield (%)[b]	Time (min)	E factor (kg waste/kg product)[c]
1	1	4-Methyl benzaldehyde(1)	**3a**	92	20	0.17
2	1	Benzadehyde(1)	**3b**	94	18	0.15

[a] All reactions are performed with 1 mol% of *p*-SAC in 1 mL of water at room temperature.

[b] Isolated yields.

[c] Exclusion of water used for work up procedure, exclusion of the amount of the *p*-SAC used, and exclusion of ethanol used for recrystallization.

Note (Regarding Table 1, SI): When the Authors have not reported the amount of solvent used in the work-up procedure, we have not accounted for solvent and considered that solvent can be recovered. By considering the *p*-SAC catalyst is recyclable and hence, waste is essentially eliminated.

For Entry 1, Table1 (SI)

E = [0.136 g (anthranilamide) + 0.120 g (4-methyl benzaldehyde) – 0.219 g (product × yield)] / 0.219 g

= 0.17

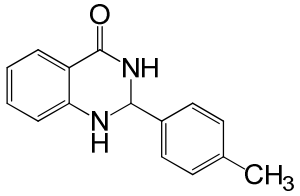
For Entry 2, Table1 (SI)

E = [0.136 g (anthranilamide) + 0.106 g (benzaldehyde) – 0.210 g (product × yield)] / 0.210 g

= 0.15

Characterization data of compounds (3):

2-(*p*-Tolyl)-2,3-dihydroquinazolin-4(1*H*)-one (3a):



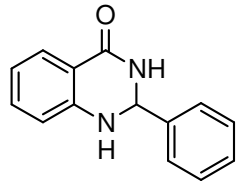
White Solid; Isolated yield: 92%; m.p. 224-225 °C;

IR: 3311, 1657, 1607, 1508, 1484, 1383 cm^{-1};

^1H NMR (400 MHz, DMSO-d_6): δ 8.24 (s, 1H), 7.63 (d, J = 8.0 Hz, 1H), 7.39 (d, J = 7.6 Hz, 2H), 7.27-7.19 (m, 3H), 7.06 (s, 1H), 6.77-6.66 (m, 2H), 5.73 (s, 1H), 2.30 (s, 3H).

^{13}C NMR (100 MHz, DMSO-d_6): δ 164.1, 148.4, 139.1, 138.2, 133.7, 129.3, 127.8, 127.3, 117.5, 115.5, 114.9, 66.9, 21.2.

2-Phenyl-2,3-dihydroquinazolin-4(1*H*)-one (3b):



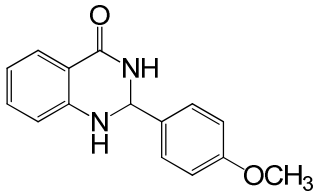
White Solid; Isolated yield: 94%; m.p. 218-220 °C;

IR: 3298, 1651, 1609, 1590, 1505, 1243 cm^{-1};

^1H NMR (400 MHz, DMSO-d_6): δ 8.30 (s, 1H), 7.64 (dd, J_1 = 8.0 Hz, J_2 = 1.6 Hz , 1H), 7.53-7.50 (m, 2H), 7.42-7.35 (m, 3H), 7.28-7.24 (m, 1H), 7.12 (s, 1H), 6.77 (d, J = 8.0 Hz, 1H), 6.71-6.67(m, 1H), 5.77 (s, 1H).

^{13}C NMR (100 MHz, DMSO-d_6): δ 164.1, 148.3, 142.1, 133.7, 128.9, 128.8 (2C), 127.8, 127.3 (2C), 117.6, 115.4, 114.9, 67.0.

2-(4-Methoxyphenyl)-2,3-dihydroquinazolin-4(1*H*)-one (3c) :



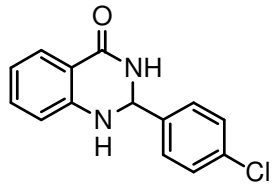
White Solid; Isolated yield: 90%; m.p. 183-184 °C;

IR: 3177, 1654, 1607, 1508, 1435, 1243, 755 cm^{-1};

^1H NMR (400 MHz, DMSO-d_6): δ 8.18 (s, 1H), 7.63-7.60 (m, 1H), 7.44-7.40 (m, 2H), 7.27-7.22 (m, 1H), 7.00-6.93 (m, 3H), 6.75-6.66 (m, 2H), 5.71 (s, 1H), 3.75 (s, 3H).

^{13}C NMR (100 MHz, DMSO-d_6): δ 164.2, 159.9, 148.5, 133.9, 133.7, 128.7 (2C), 127.8, 117.5, 115.5, 114.9, 114.1 (2C), 66.8, 55.6.

2-(4-Chlorophenyl)-2,3-dihydroquinazolin-4(1*H*)-one (3d):



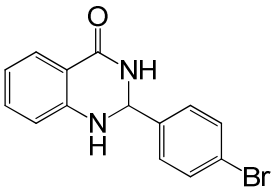
White Solid; Isolated yield: 87%; m.p. 206-208 °C;

IR: 3307, 1666, 1652, 1506, 1482 cm^{-1};

^1H NMR (400 MHz, DMSO-d_6): δ 8.34 (s, 1H), 7.62 (d, J =7.6 Hz, 1H), 7.53-7.45 (m, 4H), 7.26 (t, J =7.6 Hz, 1H), 7.15 (s, 1H), 6.77-6.67 (m, 2H), 5.78 (s, 1H).

^{13}C NMR (100 MHz, DMSO-d_6): δ 164.0, 148.1, 141.1, 133.9, 133.4, 129.2, 128.8, 127.8, 117.8, 115.4, 114.9, 66.2.

2-(4-Bromophenyl)-2,3-dihydroquinazolin-4(1*H*)-one (3e):



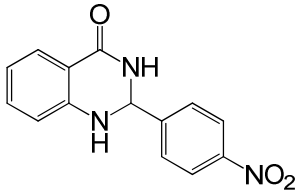
White Solid; Isolated yield: 86%; m.p. 196-198 °C;

IR: 3308, 1666, 1653, 1606, 1481 cm^{-1};

^1H NMR (400 MHz, DMSO-d_6): δ 8.34 (s, 1H), 7.63-7.59 (m, 3H), 7.45 (d, J = 8.0 Hz, 2H), 7.28-7.24 (m, 1H), 7.15 (s, 1H), 6.77-6.67 (m, 2H), 5.77 (s, 1H).

^{13}C NMR (100 MHz, DMSO-d_6): δ 164.0, 148.1, 141.6, 133.9, 131.7(2C), 129.6(2C), 127.8, 122.0, 117.8, 115.4, 114.9, 66.3.

2-(4-Nitrophenyl)-2,3-dihydroquinazolin-4(1*H*)-one (3f):



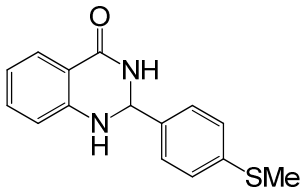
Yellow Solid; Isolated yield: 82%; m.p. 298-300 °C;

IR: 3281, 1644, 1607, 1517, 1387, 1347 cm^{-1};

^1H NMR (400 MHz, DMSO-d_6): δ 8.52 (s, 1H), 8.26 (d, *J* = 8.8 Hz, 2H), 7.75 (d, *J* = 8.8 Hz, 2H),7.63-7.60 (m, 1H), 7.33-7.25 (m, 2H), 6.79-6.68 (m, 2H), 5.92 (s, 1H).

^{13}C NMR (100 MHz, DMSO-d_6): δ 163.7, 149.7, 147.9, 147.7, 134.0, 128.5(2C), 127.9, 124.0(2C), 117.9, 115.4, 115.0, 65.8.

2-(4-(Methylthio)phenyl)-2,3-dihydroquinazolin-4(1*H*)-one (3g):



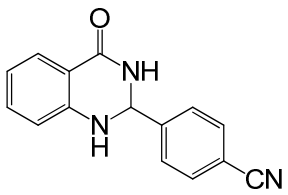
White Solid; Isolated yield: 88%; m.p. 179-180 °C;

IR: 3297, 1664, 1651, 1608, 1485, 1386, 749 cm^{-1};

^1H NMR (400 MHz, DMSO-d_6): δ 8.28 (s, 1H), 7.63 (d, *J* = 7.6 Hz, 1H), 7.44 (d, *J* = 8.0 Hz, 2H), 7.30-7.23 (m, 3H), 7.09 (s, 1H), 6.76 (d, *J* = 8.0 Hz, 1H), 6.69 (t, *J* = 7.2 Hz, 1H), 5.74 (s, 1H), 2.47 (s, 3H).

^{13}C NMR (100 MHz, DMSO-d_6): δ 164.1, 148.3, 138.9, 138.7, 133.8, 127.9(2C), 127.8, 126.2(2C), 117.6, 115.5, 114.9, 66.6, 15.2.

4-(4-Oxo-1,2,3,4-tetrahydroquinazolin-2-yl)benzonitrile (3h):



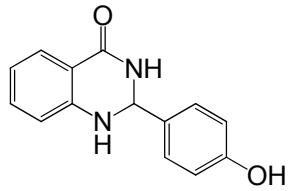
White Solid; Isolated yield: 86%; m.p. 250-251 °C;

IR: 3334, 2227, 1664, 1610, 1504, 1485, 1374, 771 cm^{-1};

^1H NMR (400 MHz, DMSO-d_6): δ 8.48 (s, 1H), 7.88 (d, J = 8.4 Hz, 2H), 7.69 -7.61 (m, 3H), 7.29-7.25 (m, 2H), 6.78 (d, J = 8.0 Hz, 1H), 6.70 (t, J = 7.2 Hz, 1H), 5.87 (s, 1H).

^{13}C NMR (100 MHz, DMSO-d_6): δ 163.8, 147.8(2C), 134.0, 132.9 (2C), 128.2 (2C), 127.9, 119.1, 117.9, 115.4, 115.0, 111.5, 66.0.

2-(4-Hydroxyphenyl)-2,3-dihydroquinazolin-4(1*H*)-one (3i):



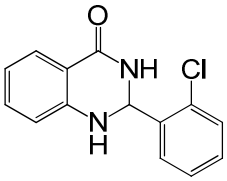
White Solid; Isolated yield: 82%; m.p. 278-280 °C;

IR: 3064, 1672, 1598, 1487, 1284, 764 cm^{-1};

^1H NMR (400 MHz, DMSO-d_6): δ 9.54 (s, 1H), 8.15 (s, 1H), 7.65-7.63 (m, 1H), 7.33 (d, J = 8.8 Hz, 2H), 7.27 -7.23 (m, 1H), 6.95 (s, 1H), 6.80-6.75 (m, 3H), 6.71-6.67 (m, 1H), 5.68 (s, 1H).

^{13}C NMR (100 MHz, DMSO-d_6): δ 164.3, 158.2, 148.6, 133.7, 132.1, 128.8(2C), 127.8, 117.5, 115.4(2C), 114.9, 67.1.

2-(2-Chlorophenyl)-2,3-dihydroquinazolin-4(1*H*)-one (3j):



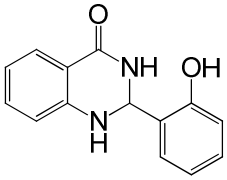
White Solid; Isolated yield: 90%; m.p. 204-205 °C;

IR: 3207, 1665, 1632, 1510, 1487 cm^{-1};

^1H NMR (400 MHz, DMSO-d_6): δ 8.22 (s, 1H), 7.69-7.66 (m, 2H), 7.52-7.49 (m, 1H), 7.42-7.40 (m, 2H), 7.29-7.25 (m, 1H), 7.02 (s, 1H), 6.79-6.71 (m, 2H), 6.16 (s, 1H).

^{13}C NMR (100 MHz, DMSO-d_6): δ 164.1, 148.1, 138.4, 133.9, 132.3, 130.8, 130.1, 129.2, 127.9 (2C), 118.0, 115.2, 115.0, 64.2.

2-(2-Hydroxyphenyl)-2,3-dihydroquinazolin-4(1*H*)-one (3k):



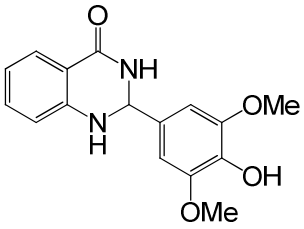
White Solid; Isolated yield: 88%; m.p. 223-224 °C;

IR: 3164, 1654, 1542, 1453, 1384, 762 cm^{-1};

^{1}H NMR (400 MHz, DMSO-d_6): δ 9.87 (s, 1H), 7.95 (s, 1H), 7.64 (d, J = 8.0 Hz, 1H), 7.36 (d, J = 7.6 Hz, 1H), 7.25 -7.14 (m, 2H), 6.88 (d, J = 8.4 Hz, 1H) 6.83-6.76 (m, 3H), 6.68 (t, J = 7.6 Hz, 1H), 6.03 (s, 1H).

^{13}C NMR (100 MHz, DMSO-d_6): δ 164.5, 155.0, 148.6, 133.7, 129.7, 128.2, 127.8, 127.6, 119.3, 117.5, 115.8, 115.2, 115.0, 61.7.

2-(4-Hydroxy-3,5-dimethoxyphenyl)-2,3-dihydroquinazolin-4(1*H*)-one (3l):



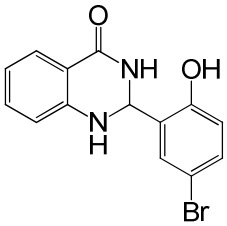
White Solid; Isolated yield: 84%; m.p. 223-224 °C;

IR: 3334, 1660, 1610, 1463, 1329, 762 cm^{-1};

^{1}H NMR (400 MHz, DMSO-d_6): δ 8.45 (s, 1H), 8.10 (s, 1H), 7.64-7.61 (m, 1H), 7.28-7.24 (m, 1H), 6.96 (s, 1H), 6.80-6.67 (m, 4H), 5.67 (s, 1H), 3.76 (s, 6H).

^{13}C NMR (100 MHz, DMSO-d_6): δ 164.3, 148.7, 148.2(2C), 136.4, 133.7, 131.3, 127.8, 117.6, 115.5, 114.9, 105.2 (2C), 67.6, 56.4 (2C).

2-(5-Bromo-2-hydroxyphenyl)-2,3-dihydroquinazolin-4(1*H*)-one (3m):



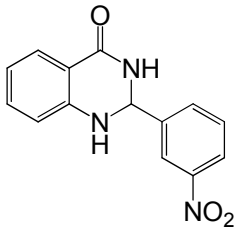
White Solid; Isolated yield: 85%; m.p. 262-264 °C;

IR: 3198, 1638, 1613, 1486, 1247, 751 cm^{-1};

^1H NMR (400 MHz, DMSO-d_6): δ 10.25 (s, 1H), 8.03 (s, 1H), 7.64 (d, *J* = 7.6 Hz, 1H), 7.42 (d, *J* = 2.4 Hz, 1H), 7.35 -7.32 (m, 1H), 7.27-7.23 (m, 1H), 6.86-6.78 (m, 3H), 6.70 (t, *J* = 7.6 Hz, 1H), 5.98 (s, 1H).

^{13}C NMR (100 MHz, DMSO-d_6): δ 164.3, 154.5, 148.3, 133.8, 132.3, 130.2(2C), 127.8, 118.1, 117.7, 115.1, 115.0, 110.3, 61.4.

2-(3-Nitrophenyl)-2,3-dihydroquinazolin-4(1*H*)-one (3n):



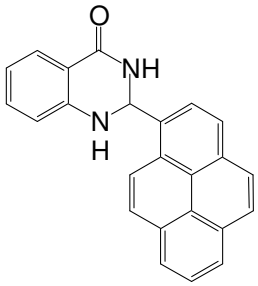
Yellow Solid; Isolated yield: 88%; m.p. 196-198 °C;

IR: 3288, 1652, 1612, 1487, 1342 cm^{-1};

^1H NMR (400 MHz, DMSO-d_6): δ 8.54 (s, 1H), 8.38 (s, 1H), 8.22-8.20 (m, 1H), 7.96 (d, *J* = 7.6 Hz, 1H), 7.70 (t, *J* = 8.0 Hz, 1H), 7.63(d, *J* = 7.6 Hz, 1H), 7.35 (s, 1H), 7.30-7.26 (m, 1H), 6.80 (d, *J* = 8.0 Hz, 1H) 6.71 (t, *J* = 7.6 Hz, 1H), 5.96 (s, 1H).

^{13}C NMR (100 MHz, DMSO-d_6): δ 163.8, 148.2, 147.8, 144.8, 134.1, 133.8, 130.5, 127.9, 123.7, 122.0, 118.0, 115.4, 115.1, 65.7.

2-(Pyren-1-yl)-2,3-dihydroquinazolin-4(1*H*)-one (3o):



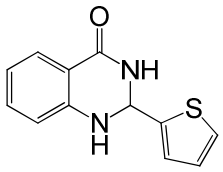
Yellow Solid; Isolated yield: 82%; m.p. 196-198 °C;

IR: 3402, 1662, 1612, 1484, 1390, 833 cm^{-1};

^{1}H NMR (400 MHz, DMSO-d_6): δ 8.83 (d, J = 9.6 Hz, 1H), 8.44 (s, 1H), 8.36-8.34 (m, 4H), 8.29 (d, J = 9.2 Hz, 1H), 8.24 (d, J = 2.8 Hz, 2H), 8.12 (t, J = 7.6 Hz, 1H), 7.81-7.78 (m, 1H), 7.33-7.30 (m, 1H), 7.25 (s, 1H), 6.90 (s, 1H), 6.85-6.78 (m, 2H).

^{13}C NMR (100 MHz, DMSO-d_6): δ 164.6, 149.1, 133.8, 133.5, 131.7, 131.2, 130.6, 128.9, 128.3, 128.1, 128.0, 127.8, 126.9, 126.6, 126.0, 125.9, 125.2, 124.7, 124.4, 124.1, 117.9, 115.7, 115.1, 66.0.

2-(Thiophen-2-yl)-2,3-dihydroquinazolin-4(1*H*)-one(3p):



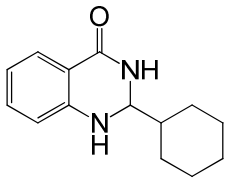
White Solid; Isolated yield: 86%; m.p. 223-224 °C;

IR: 3287, 1650, 1607, 1515, 1487, 763 cm^{-1};

^{1}H NMR (400 MHz, DMSO-d_6): δ 8.46 (s, 1H), 7.64 (d, J = 8.0 Hz, 1H), 7.46 (d, J = 4.8 Hz, 1H),7.29-7.25 (m, 2H), 7.14 (d, J = 2.8 Hz, 1H), 6.99 (t, J = 3.6 Hz, 1H), 6.79 -6.70 (m, 2H), 6.03 (s, 1H).

^{13}C NMR (100 MHz, DMSO-d_6): δ 163.6, 147.7, 146.9, 133.8, 127.8, 126.9, 126.4, 126.2, 118.0, 115.6, 115.2, 63.0.

2-Cyclohexyl-2,3-dihydroquinazolin-4(1*H*)-one(3q):



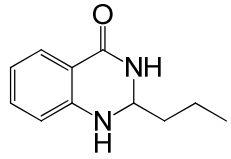
White Solid; Isolated yield: 82%; m.p. 223-224 °C;

IR: 3173, 2924, 1644, 1609, 1505, 1070, 751 cm^{-1};

^{1}H NMR (400 MHz, DMSO-d_6): δ 7.87 (s, 1H), 7.56 (d, J = 7.6 Hz, 1H), 7.20 (t, J = 8.0 Hz, 1H), 6.74 (d, J = 8.0 Hz, 1H), 6.63 -6.55 (m, 2H), 4.45 (s, 1H), 1.72-1.56 (m, 6H), 1.16-1.12 (m, 5H).

^{13}C NMR (100 MHz, DMSO-d_6): δ 164.2, 148.8, 133.5, 127.7, 126.9, 116.9, 115.2, 114.5, 69.0, 43.3, 27.5, 27.1, 26.4, 26.0.

2-Propyl-2,3-dihydroquinazolin-4(1*H*)-one(3r):



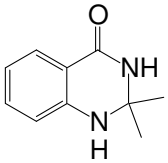
White Solid; Isolated yield: 68%; m.p. 149-150 °C;

IR: 2911, 1642, 1607, 1515, 1483, 748 cm^{-1};

^1H NMR (400 MHz, $CDCl_3$): δ 7.91-7.9 (m, 1H), 7.34-7.29 (m, 1H), 6.87 (t, *J* = 7.2 Hz, 1H), 6.69 (d, *J* = 8.0 Hz, 2H), 4.91 (t, *J* = 5.6 Hz, 1H), 1.82 -1.77 (m, 2H), 1.55-1.48 (m, 2H), 1.02 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 165.6, 147.5, 133.8, 128.5, 119.3, 116.0, 114.7, 65.1, 37.6, 17.4, 13.8.

2,2-Dimethyl-2,3-dihydroquinazolin-4(1*H*)-one(3s):



White Solid; Isolated yield: 64%; m.p. 185-186 °C;

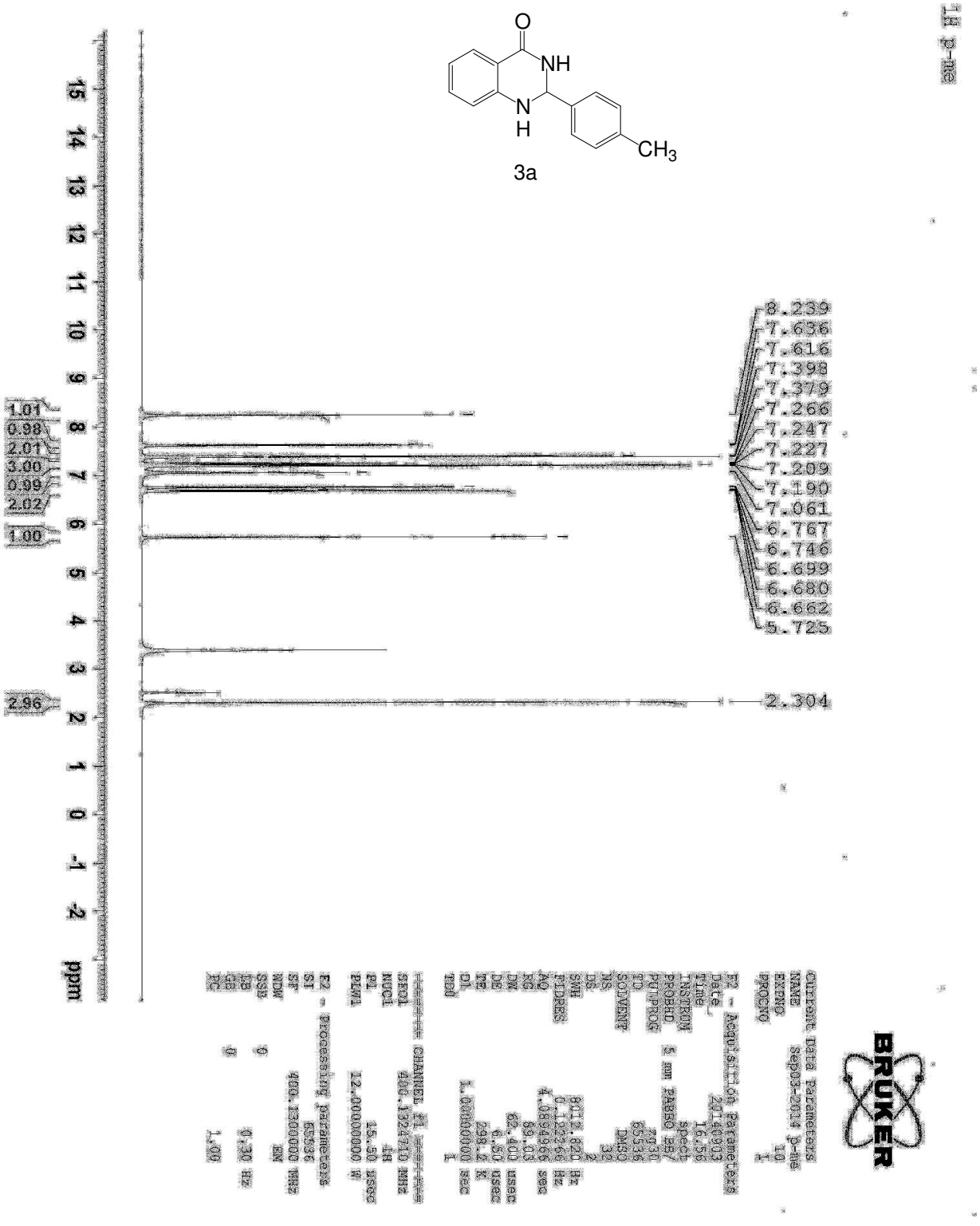
IR: 3211, 1645, 1598, 1510, 1487 cm^{-1};

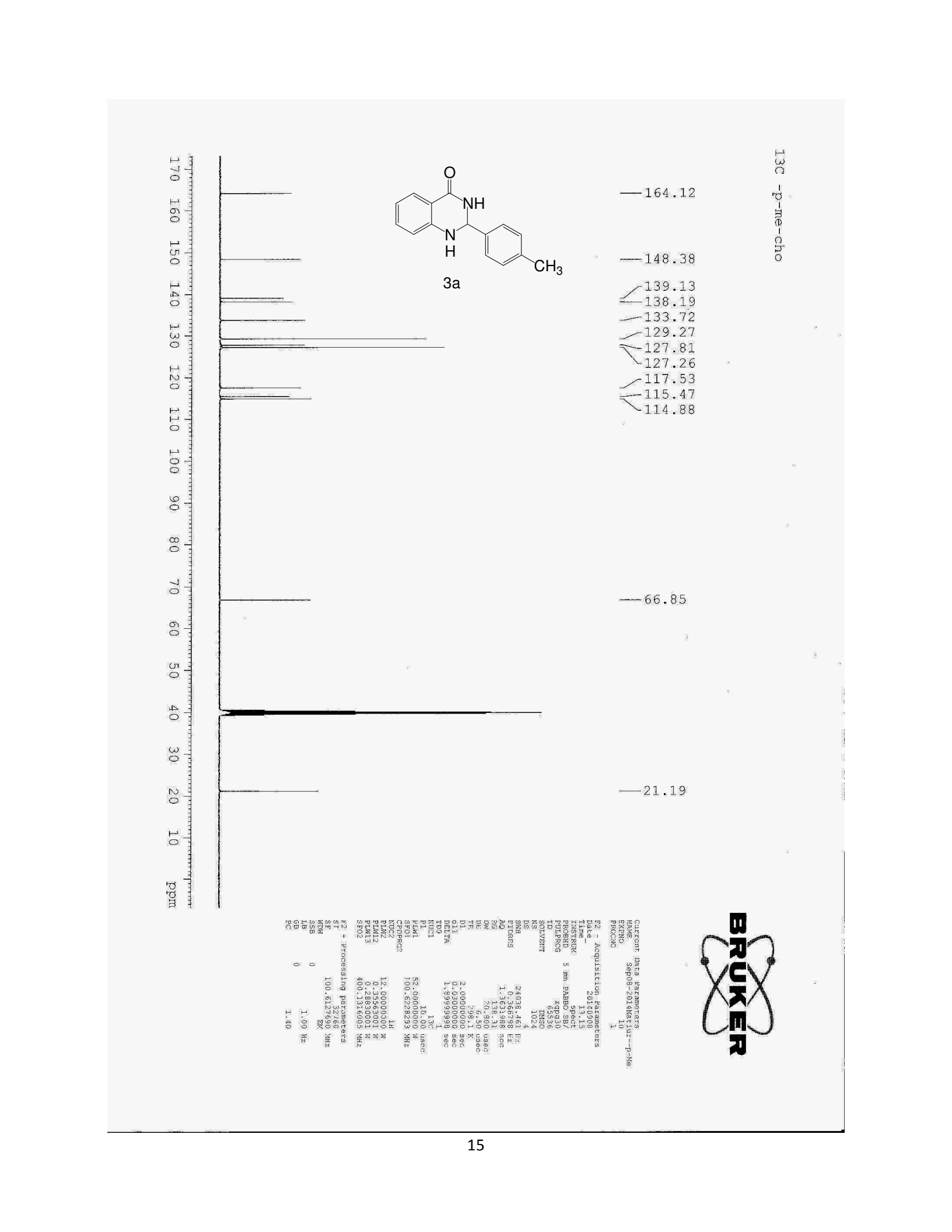
^1H NMR (400 MHz, DMSO-d_6): δ 7.94 (s, 1H), 7.59(d, *J* = 7.6 Hz, 1H), 7.24 -7.20 (m, 1H), 6.64-6.63 (m, 3H), 1.39 (s, 6H).

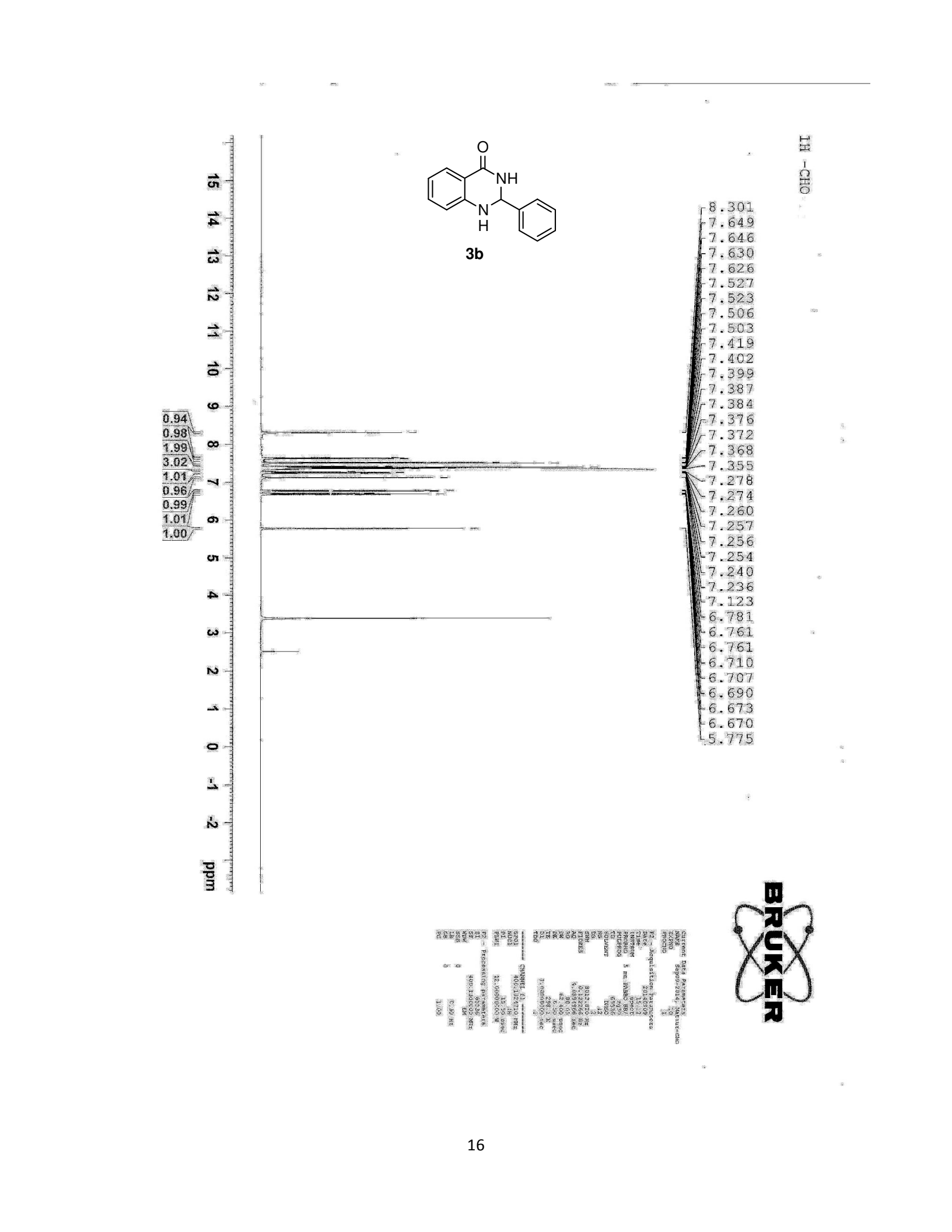
^{13}C NMR (100 MHz, DMSO-d_6): δ 163.5, 147.5, 133.7, 127.6, 116.9, 114.7, 114.3, 67.3, 29.5(2C).

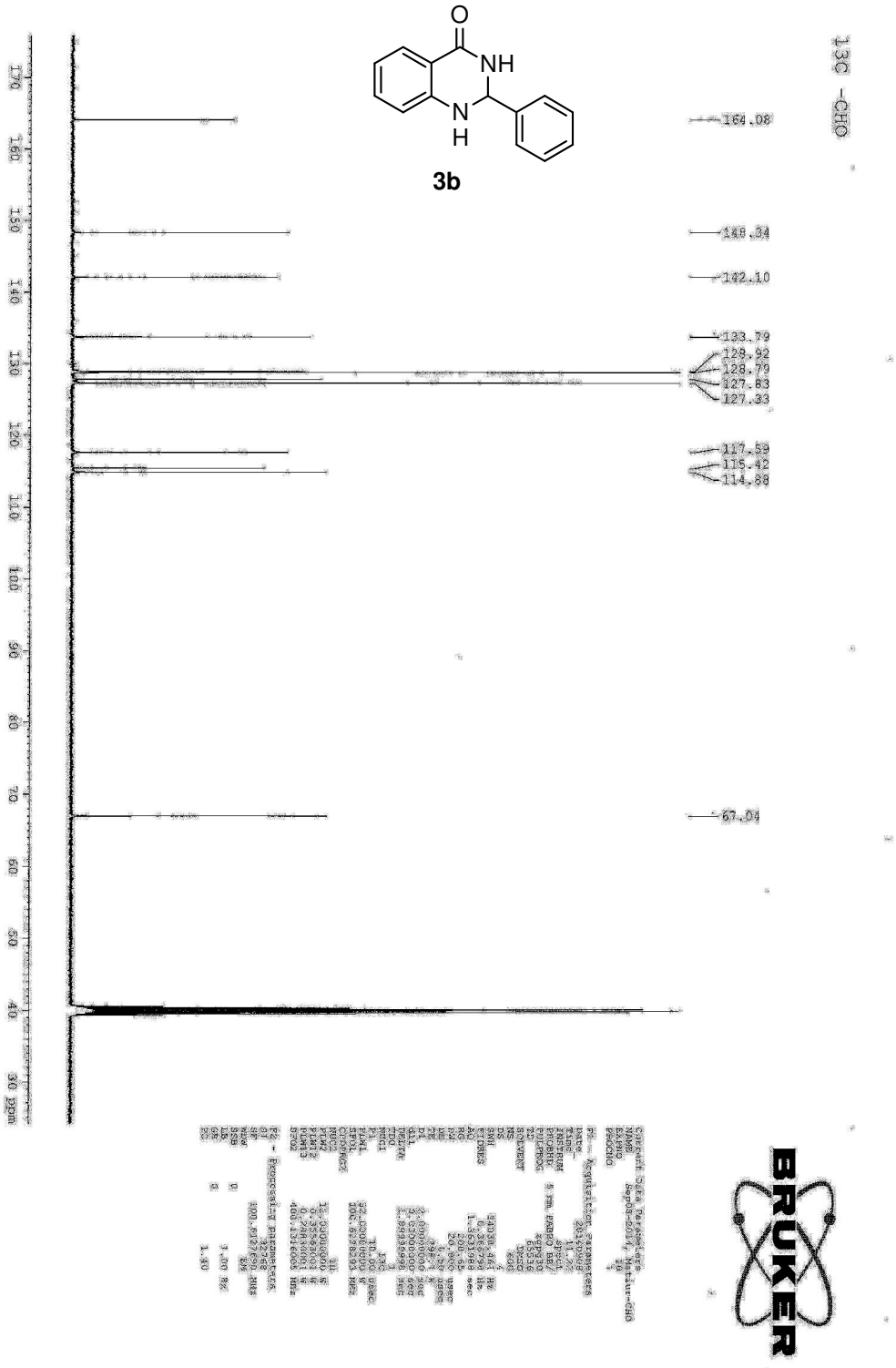
Reference:

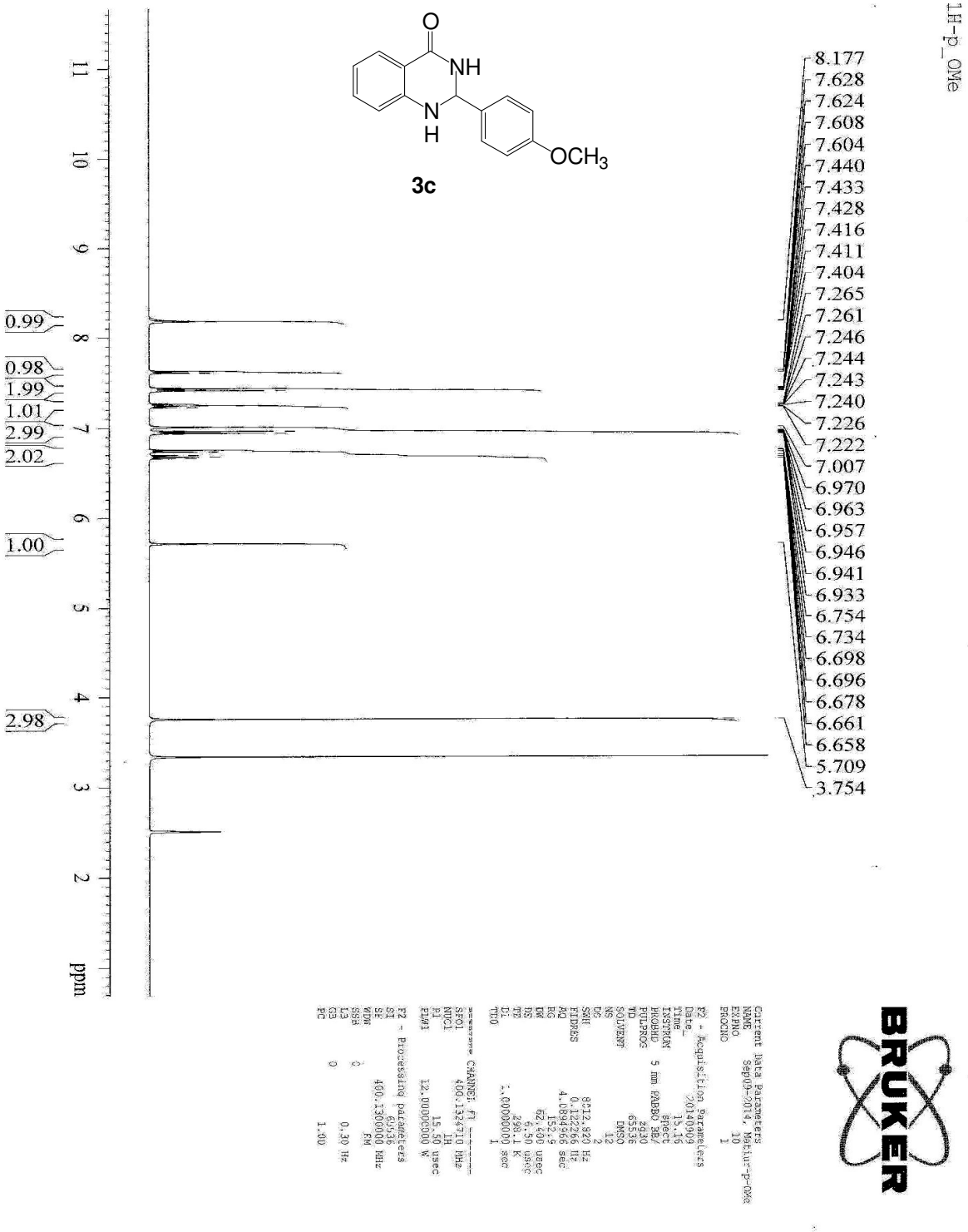
1. A. Kamal, V. Srinivasulu, B. N. Seshadri, N. Markandeya, A. Alarifi and N. Shankaraiah, *Green Chem.* 2012, **14**, 2513.


1H p-me
O
NH
N
H
CH3
3a
8.239
7.636
7.616
7.398
7.379
7.266
7.247
7.227
7.209
7.190
7.061
6.767
6.746
6.699
6.680
6.662
5.725
2.304
1.01
0.98
2.01
3.00
0.99
2.02
1.00
2.96
15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 ppm
Current Data Parameters
NAME p-me
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ Sep03-2014
Time 16.56
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 32
DS 2
SWH 8012.820 Hz
FIDRES 0.122268 Hz
AQ 4.0894966 sec
RG 59.03
DW 62.400 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 15.50 usec
PLW1 12.00000000 W
F2 - Processing parameters
SI 65536
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

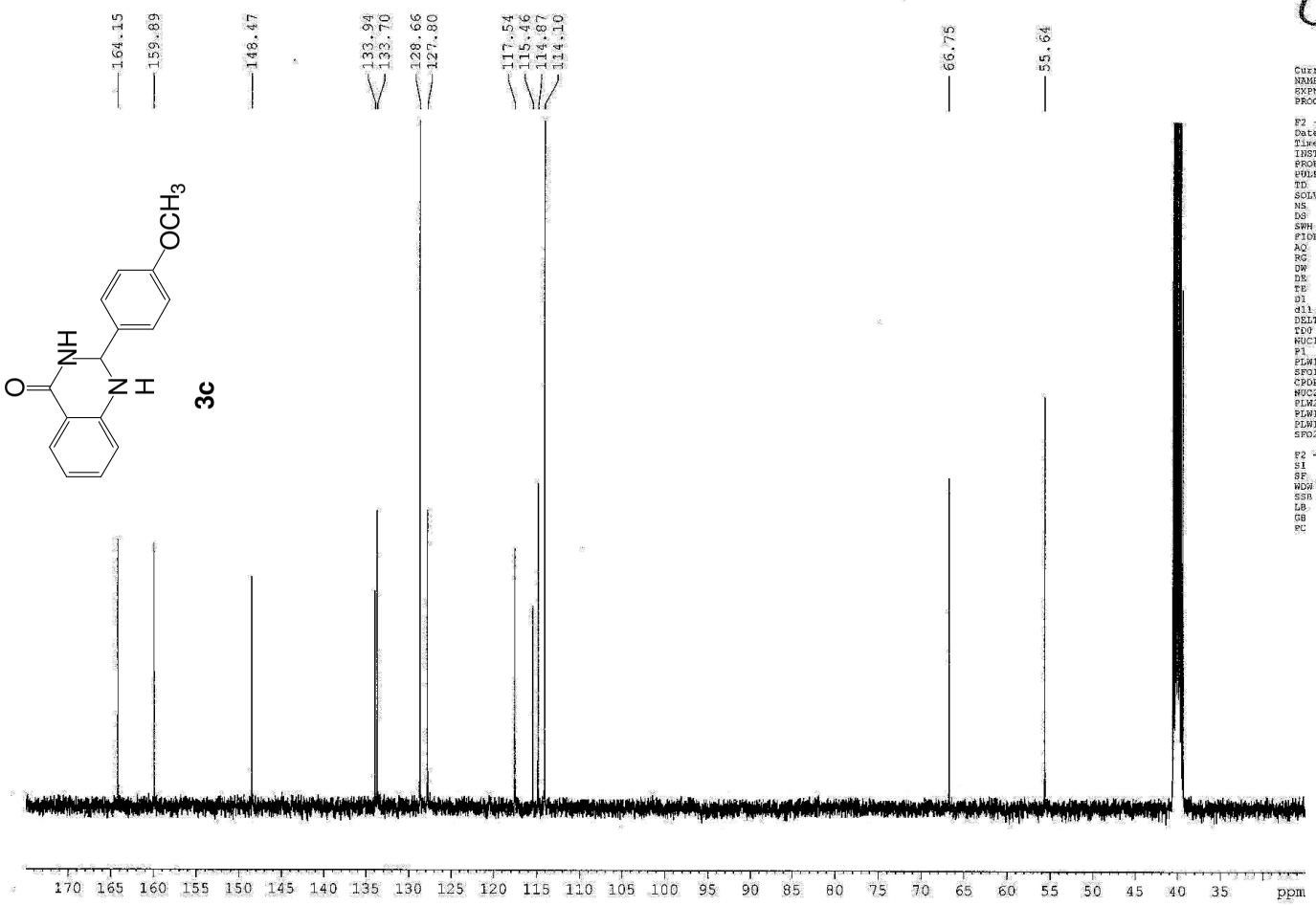

13C -p-me-cho
O
NH
N
H
CH3
3a
164.12
148.38
139.13
138.19
133.72
129.27
127.81
127.26
117.53
115.47
114.88
66.85
21.19
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
BRUKER
Current Data Parameters
NAME 3ap08-2014mar1ur-p-Me
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140908
Time_ 13.15
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 138.31
DW 20.800 usec
DE 6.50 usec
TE 298.1 K
D1 2.00000000 sec
D11 0.03000000 sec
DELTA 1.89999999 sec
TD0 1
NUC1 13C
P1 10.00 usec
PL1 52.00000000 W
SFO1 100.6228293 MHz
CPDPRG2 waltz16
NUC2 1H
PCPD2 12.00000000 W
PL2 0.13569001 W
PL12 0.28830001 W
PL13 0.28830001 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


1H -CHO
3b
8.301
7.649
7.646
7.630
7.626
7.527
7.523
7.506
7.503
7.419
7.402
7.399
7.387
7.384
7.376
7.372
7.368
7.355
7.278
7.274
7.260
7.257
7.256
7.254
7.240
7.236
7.123
6.781
6.761
6.761
6.710
6.707
6.690
6.673
6.670
5.775
0.94
0.98
1.99
3.02
1.01
0.96
0.99
1.01
1.00
ppm
BRUKER


O
NH
N
H
3b
13C -CHO
164.08
148.34
142.10
133.79
129.92
128.79
127.83
127.33
117.59
115.42
114.88
67.04
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30 ppm
BRUKER


1H-p_OMe
3c
8.177
7.628
7.624
7.608
7.604
7.440
7.433
7.428
7.416
7.411
7.404
7.265
7.261
7.246
7.244
7.243
7.240
7.226
7.222
7.007
6.970
6.963
6.957
6.946
6.941
6.933
6.754
6.734
6.698
6.696
6.678
6.661
6.658
5.709
3.754
0.99
0.98
1.99
1.01
2.99
2.02
1.00
2.98
ppm
Current Data Parameters
NAME
EXPNO
PROCNO
F2 - Acquisition Parameters
Date_
Time
INSTRUM
PROBHD
PULPROG
TD
SOLVENT
NS
DS
SWH
FIDRES
AQ
RG
DW
DE
TE
D1
TD0
F2 - Processing parameters
SI
SF
WDW
SSB
LB
GB
PC

13C -p-OMe

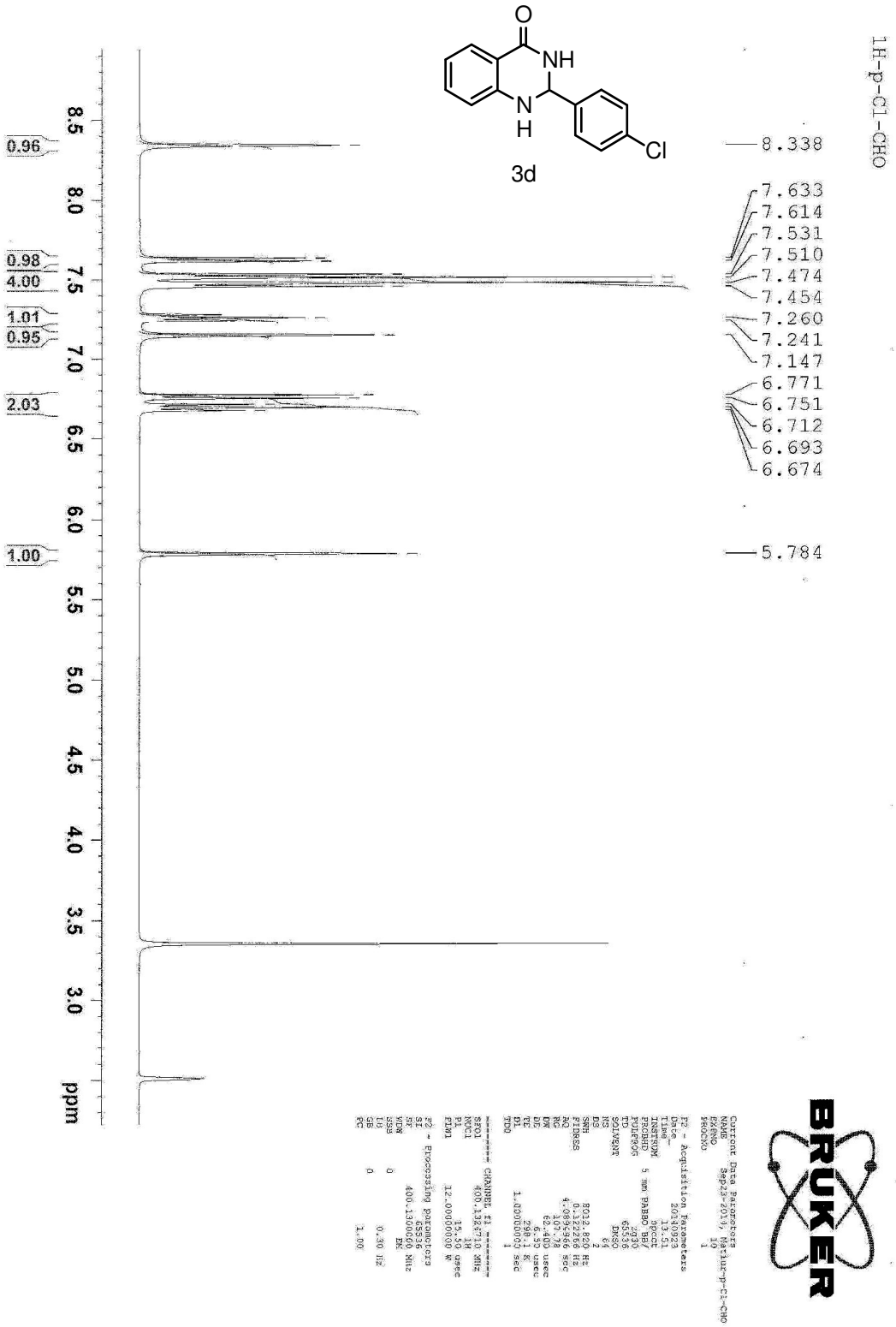


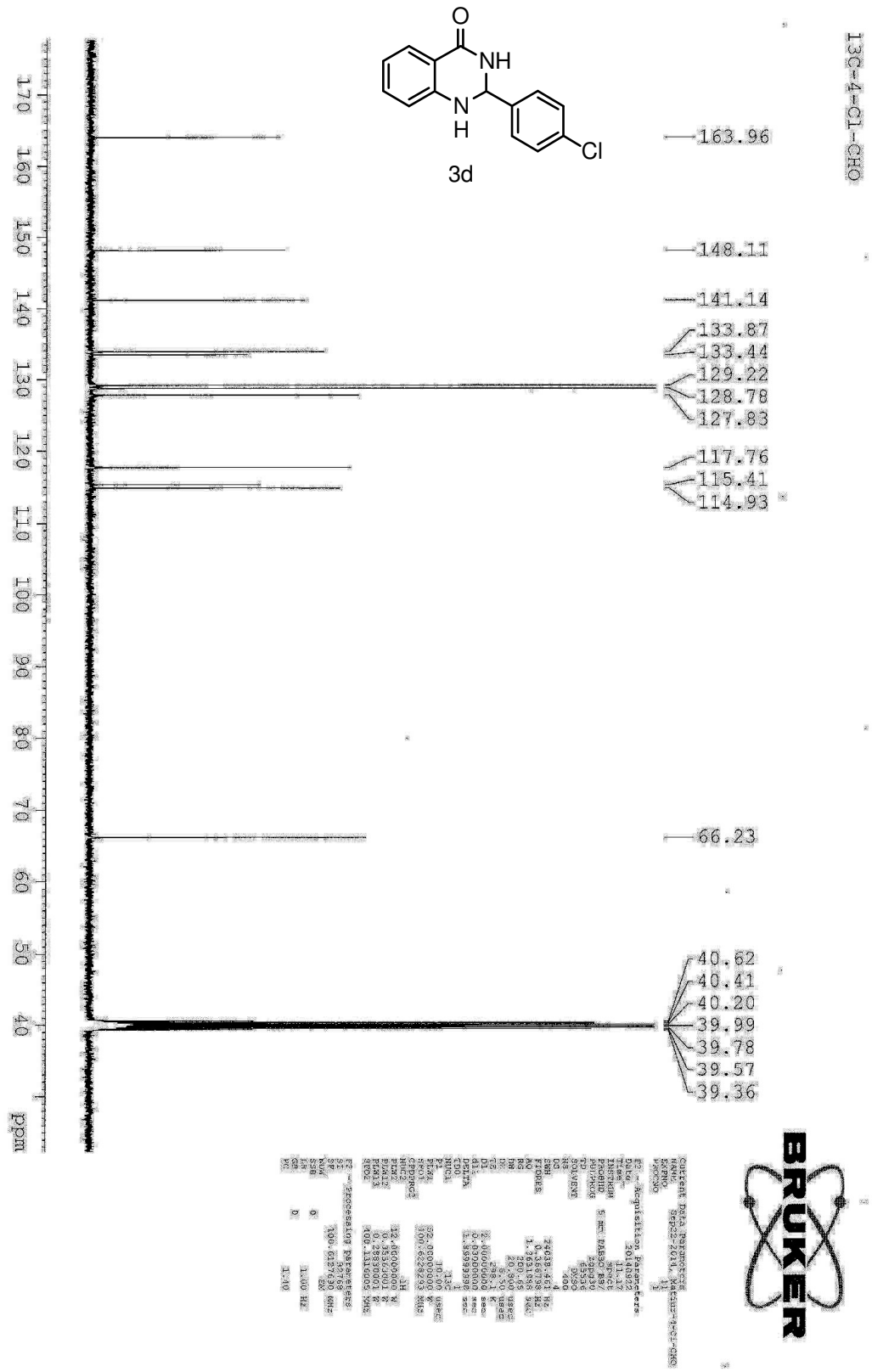
BRUKER

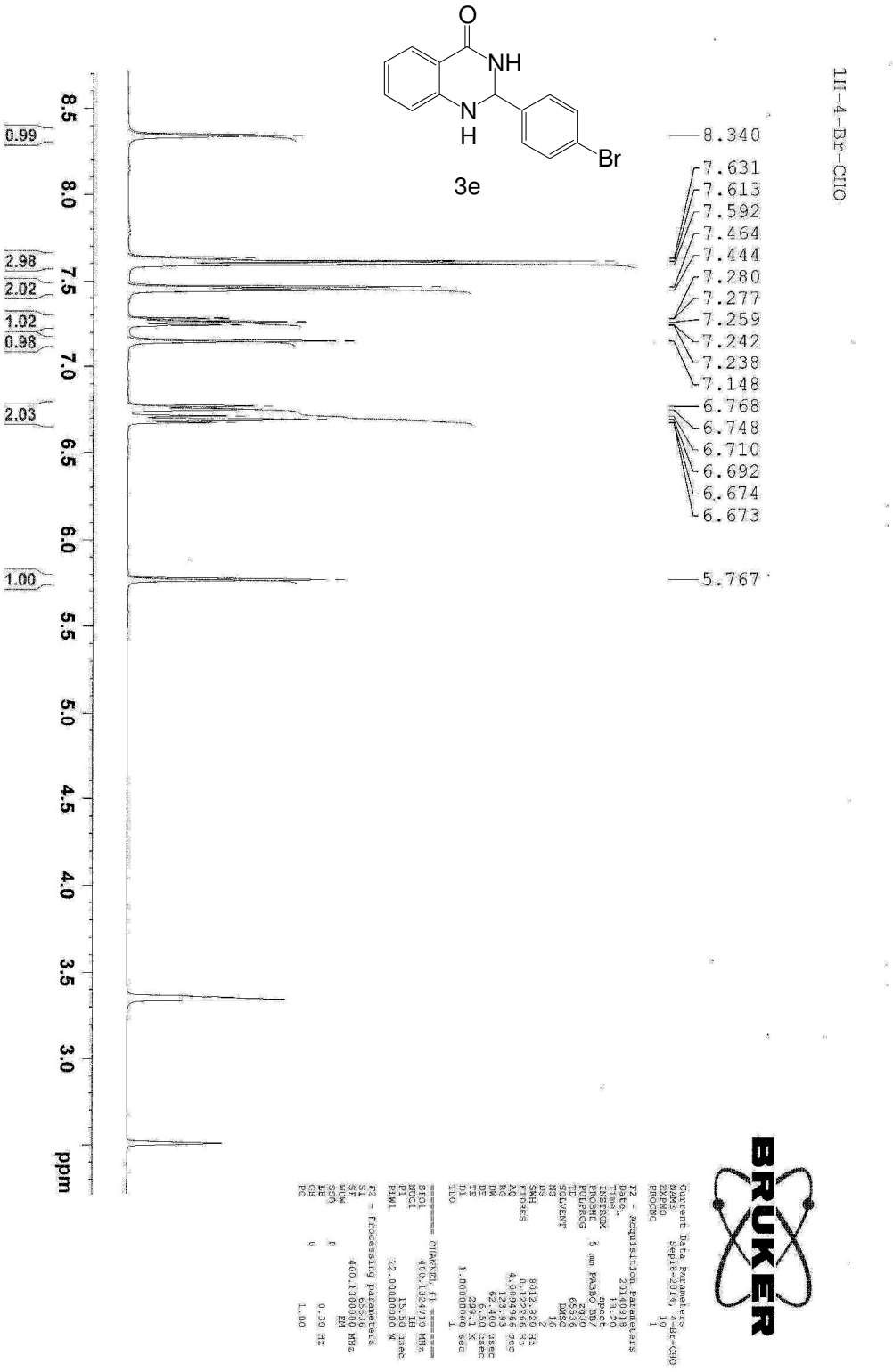
Current Data Parameters
NAME Sep08-2014, Natiur-p-OMe
EXPNO 10
PROCNO 1

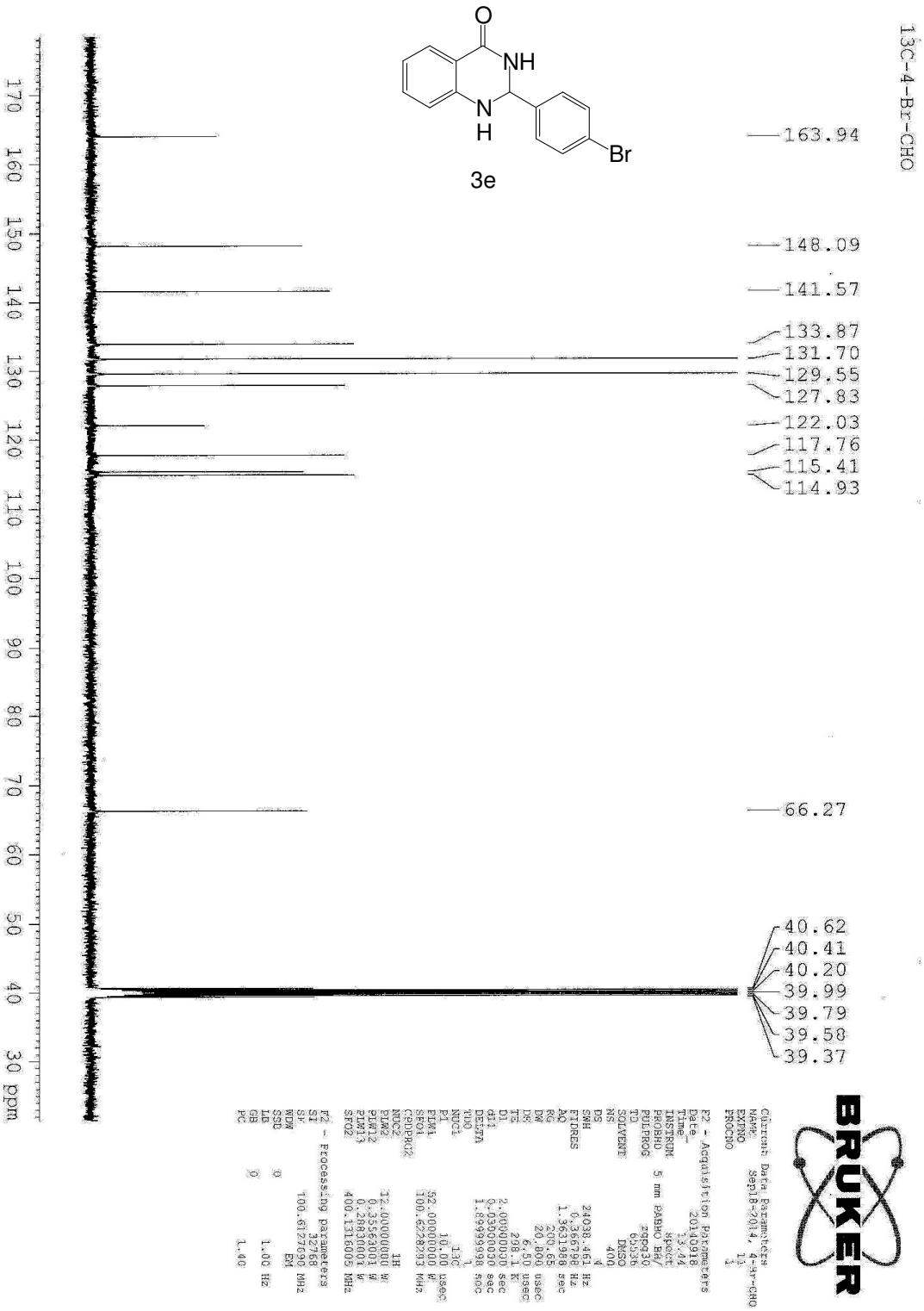
F2 - Acquisition Parameters
Date_ 20140908
Time 11.59
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 600
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 138.31
DW 20.800 usec
DE 6.50 usec
TE 298.1 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
NUC1 13C
P1 10.00 usec
PLW1 52.00000000 W
SFO1 100.6228293 MHz
CPDPRG2
NUC2 1H
PLW2 12.00000000 W
PLW12 0.35563001 W
PLW13 0.28830001 W
SFO2 400.1316005 MHz

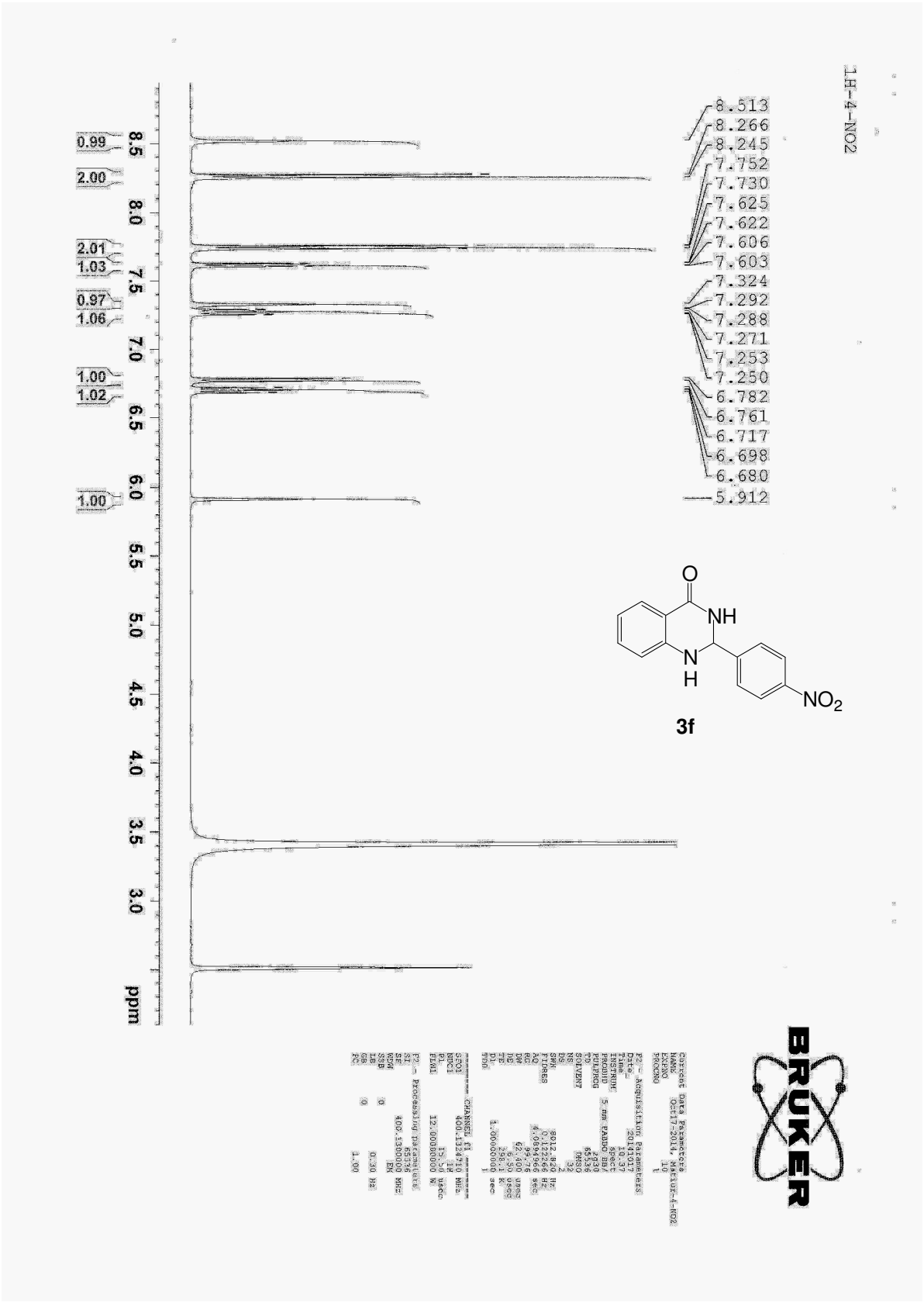
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

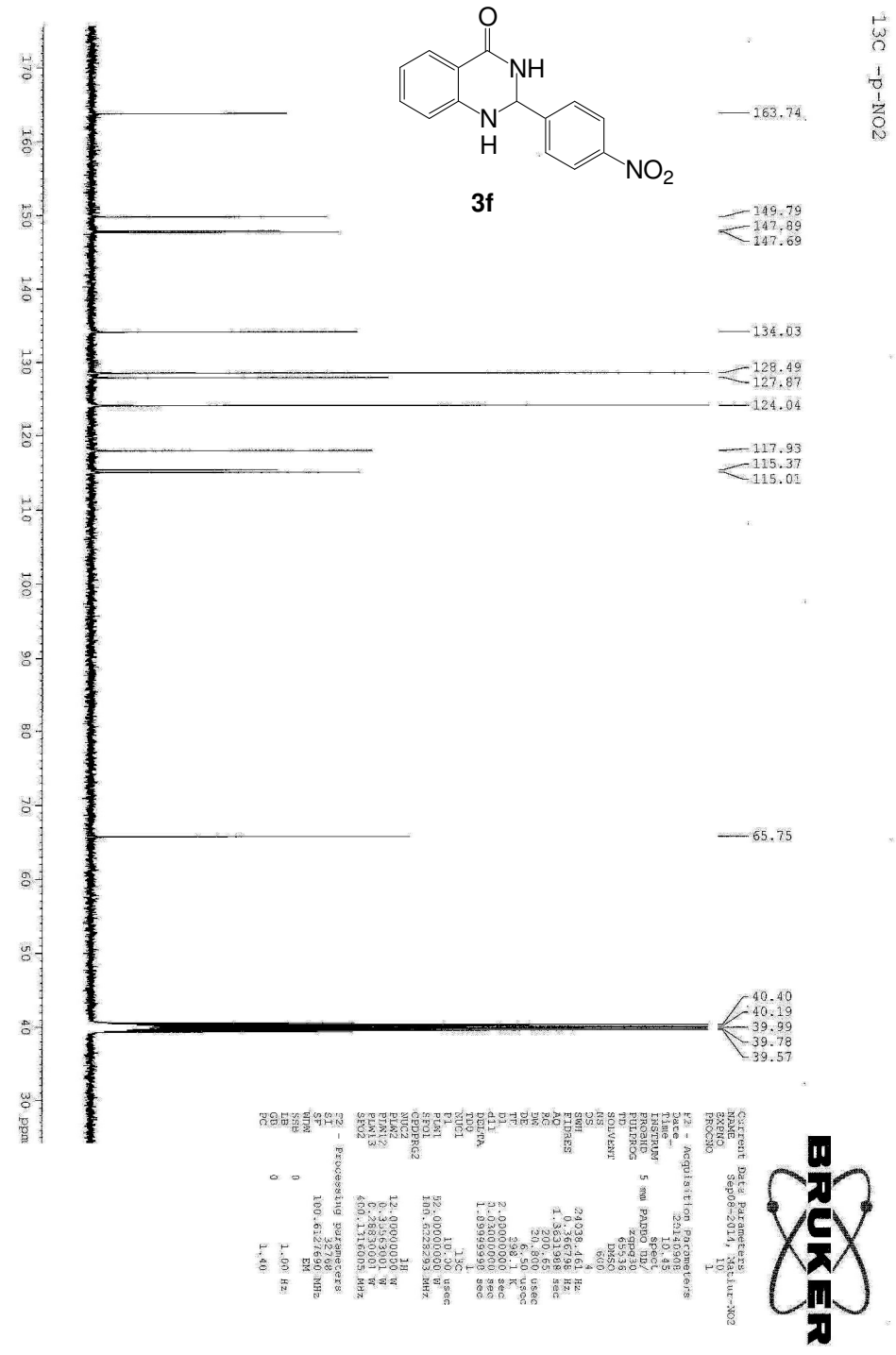

1H-p-Cl-CHO
3d
8.338
7.633
7.614
7.531
7.510
7.474
7.454
7.260
7.241
7.147
6.771
6.751
6.712
6.693
6.674
5.784
0.96
0.98
4.00
1.01
0.95
2.03
1.00
ppm

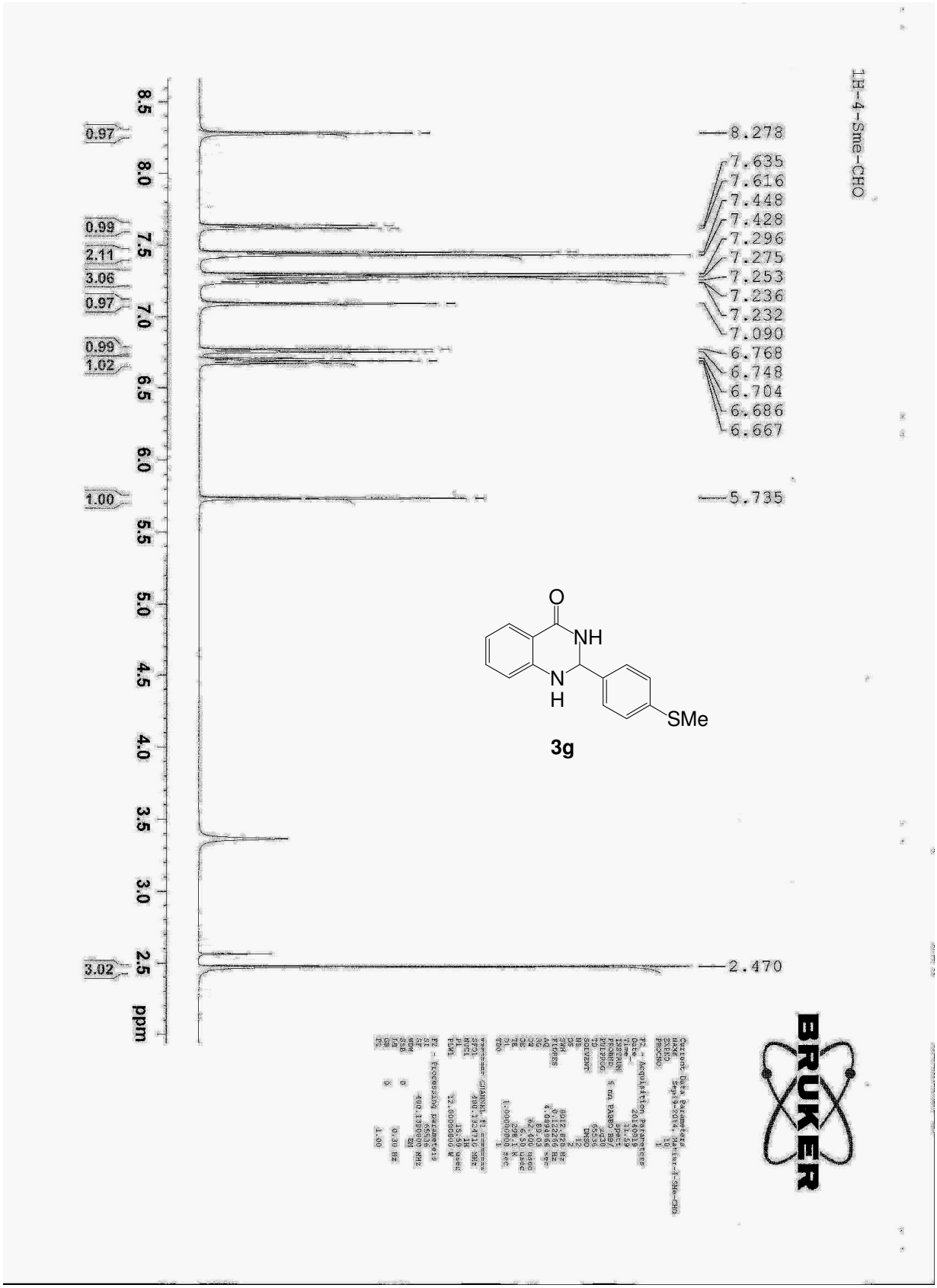

13C-4-Cl-CHO
3d
163.96
148.11
141.14
133.87
133.44
129.22
128.78
127.83
117.76
115.41
114.93
66.23
40.62
40.41
40.20
39.99
39.78
39.57
39.36
ppm
BRUKER

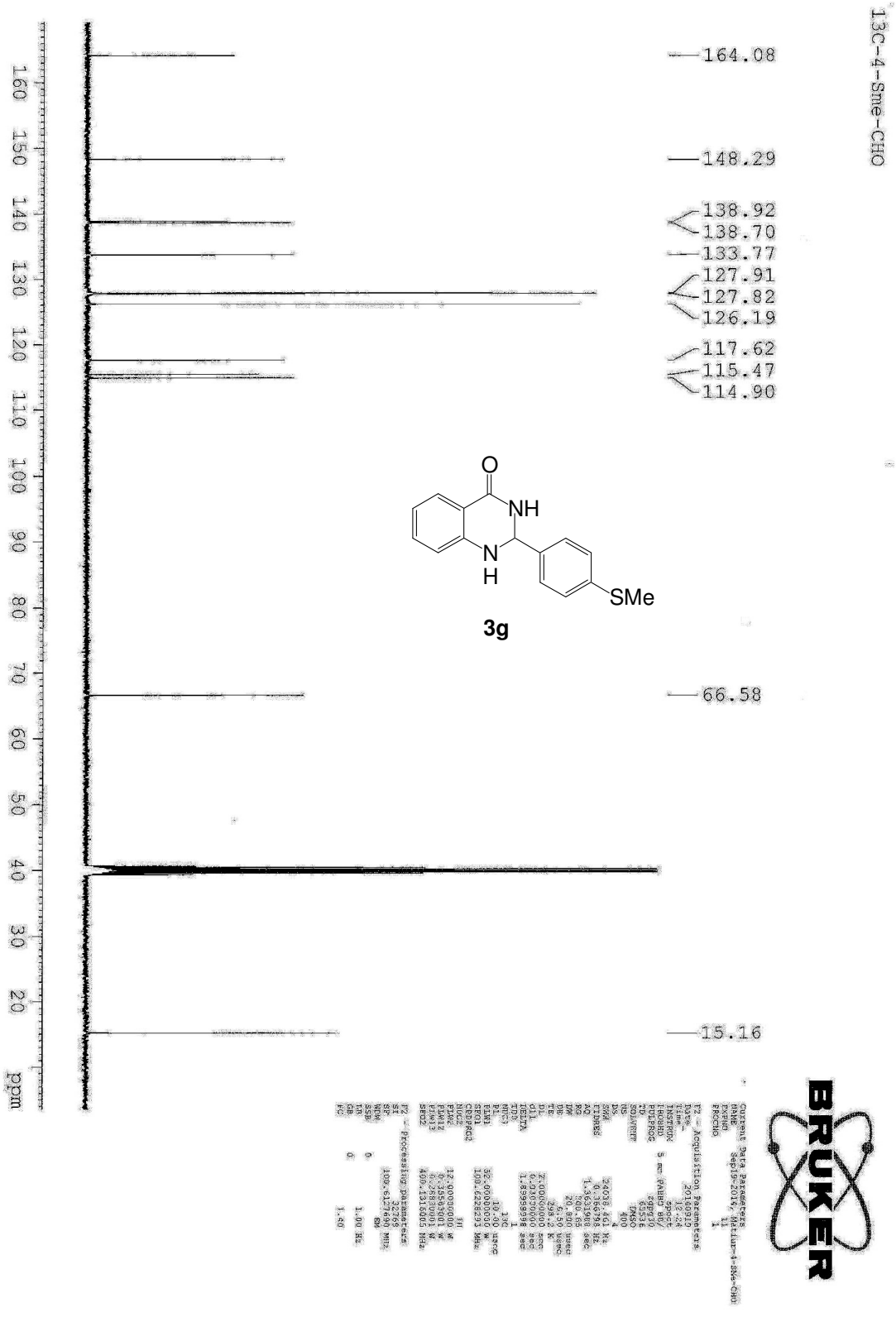

1H-4-Br-CHO
3e
8.340
7.631
7.613
7.592
7.464
7.444
7.280
7.277
7.259
7.242
7.238
7.148
6.768
6.748
6.710
6.692
6.674
6.673
5.767
0.99
2.98
2.02
1.02
0.98
2.03
1.00
ppm

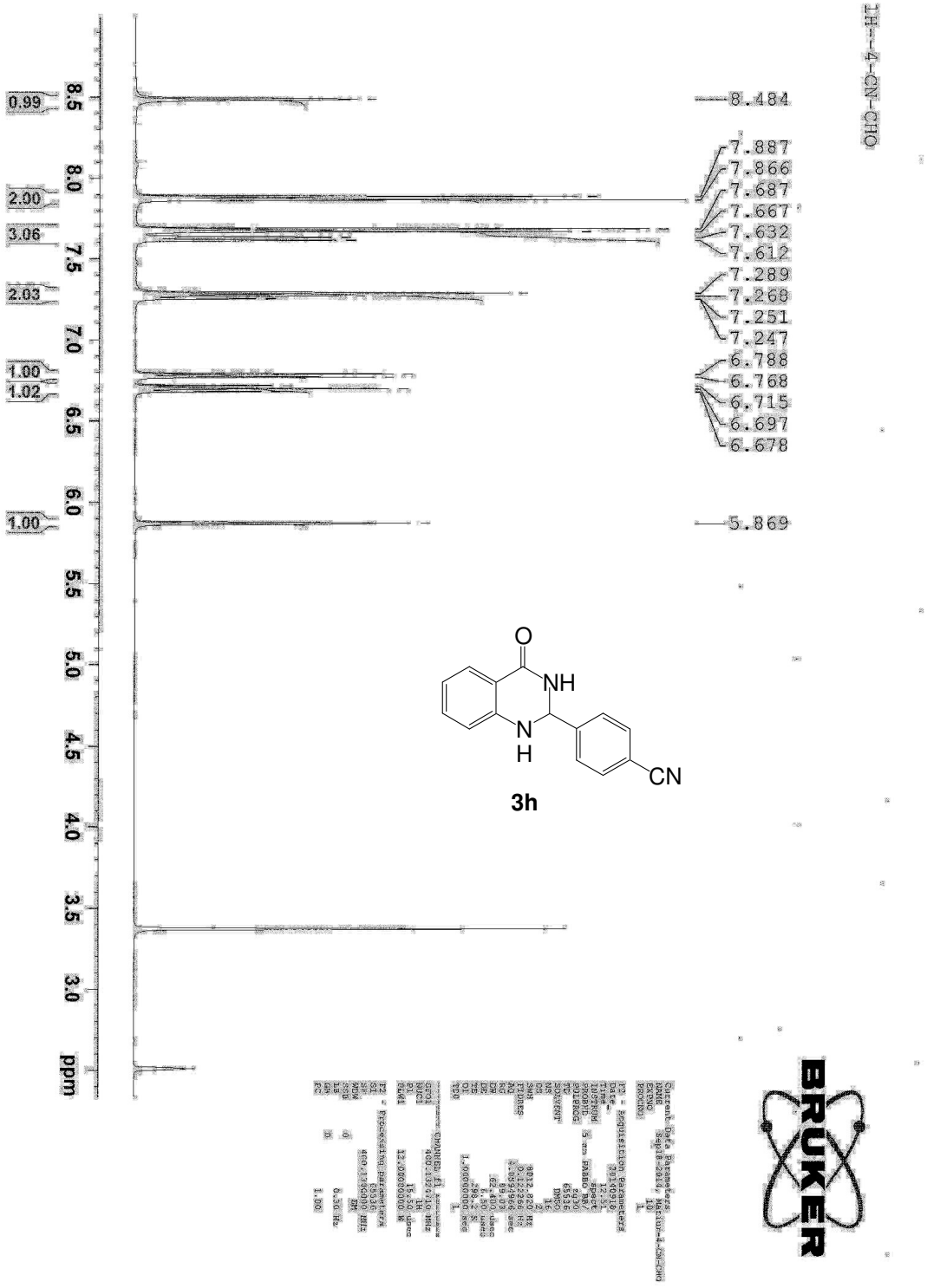

13C-4-Br-CHO
O
NH
N
H
Br
3e
163.94
148.09
141.57
133.87
131.70
129.55
127.83
122.03
117.76
115.41
114.93
66.27
40.62
40.41
40.20
39.99
39.79
39.58
39.37
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 ppm
Current Data Parameters
NAME Sep18-2014, 4-Br-CHO
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140918
Time 13.44
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 400
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631968 sec
RG 200.65
DW 20.800 usec
DE 6.50 usec
TE 298.1 K
D1 2.00000000 sec
dif 0.03000000 sec
DE2FA 1.89999998 sec
NUC1 1 13C
P1 10.00 usec
PL1 52.00000000 W
SFO1 100.6228293 MHz
CPDPRG2
NUC2 1H
PLW2 12.00000000 W
PLW12 0.35563001 W
PLW13 0.20830001 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
BRUKER

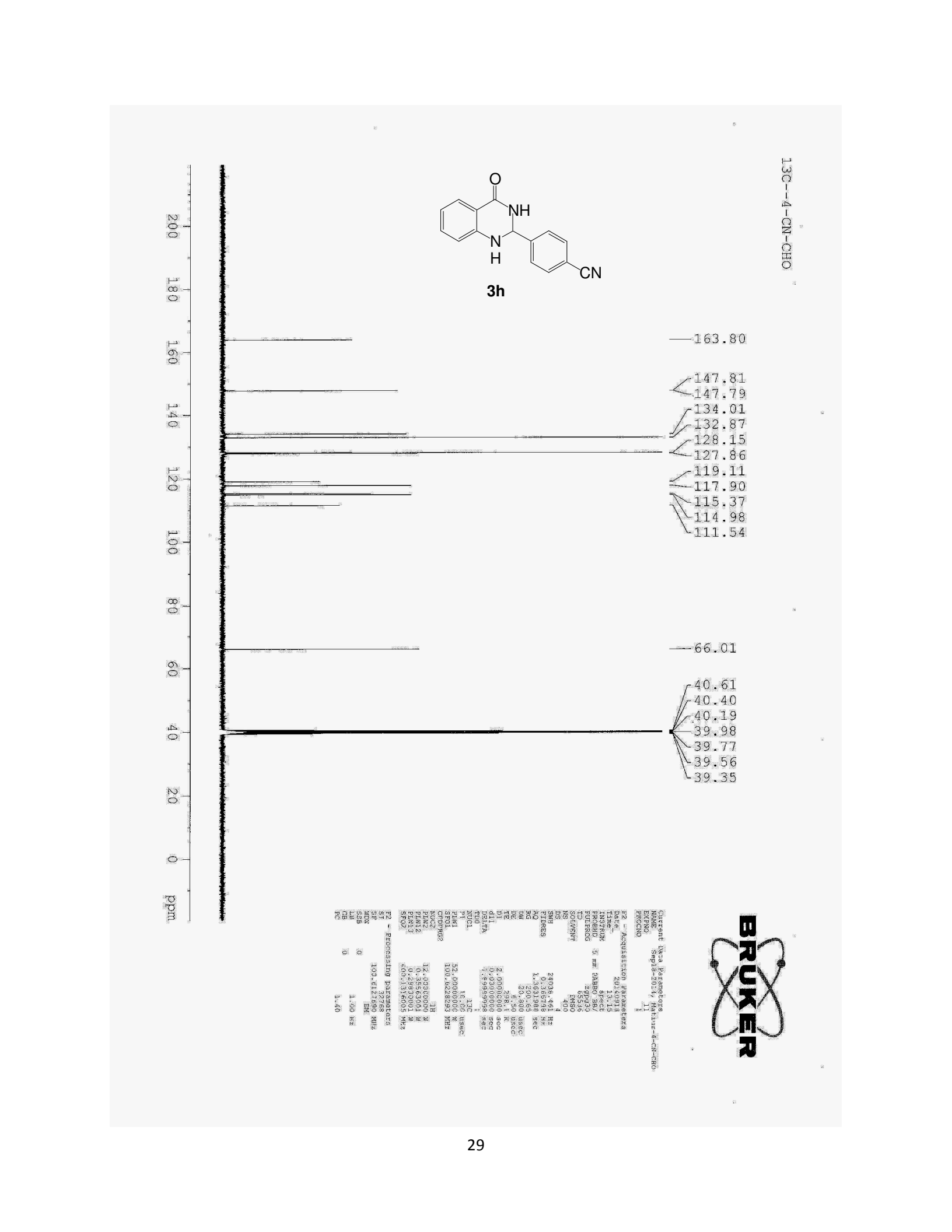

1H-4-NO2
8.513
8.266
8.245
7.752
7.730
7.625
7.622
7.606
7.603
7.324
7.292
7.288
7.271
7.253
7.250
6.782
6.761
6.717
6.698
6.680
5.912
0.99
2.00
2.01
1.03
0.97
1.06
1.00
1.02
1.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
ppm
O
NH
N
H
NO2
3f
BRUKER

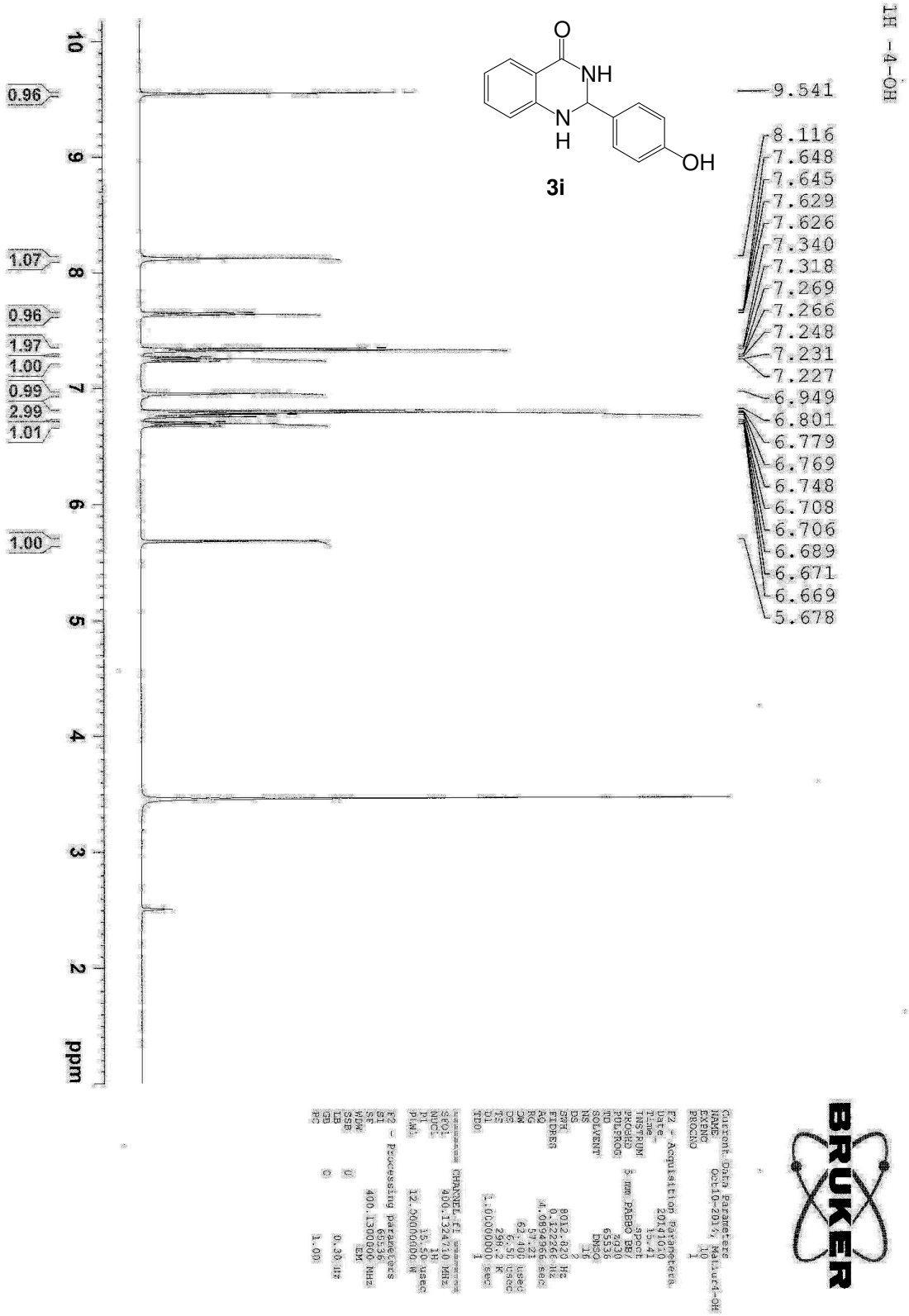

13C -p-NO2
3f
163.74
149.79
147.89
147.69
134.03
128.49
127.87
124.04
117.93
115.37
115.01
65.75
40.40
40.19
39.99
39.78
39.57
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30 ppm
BRUKER

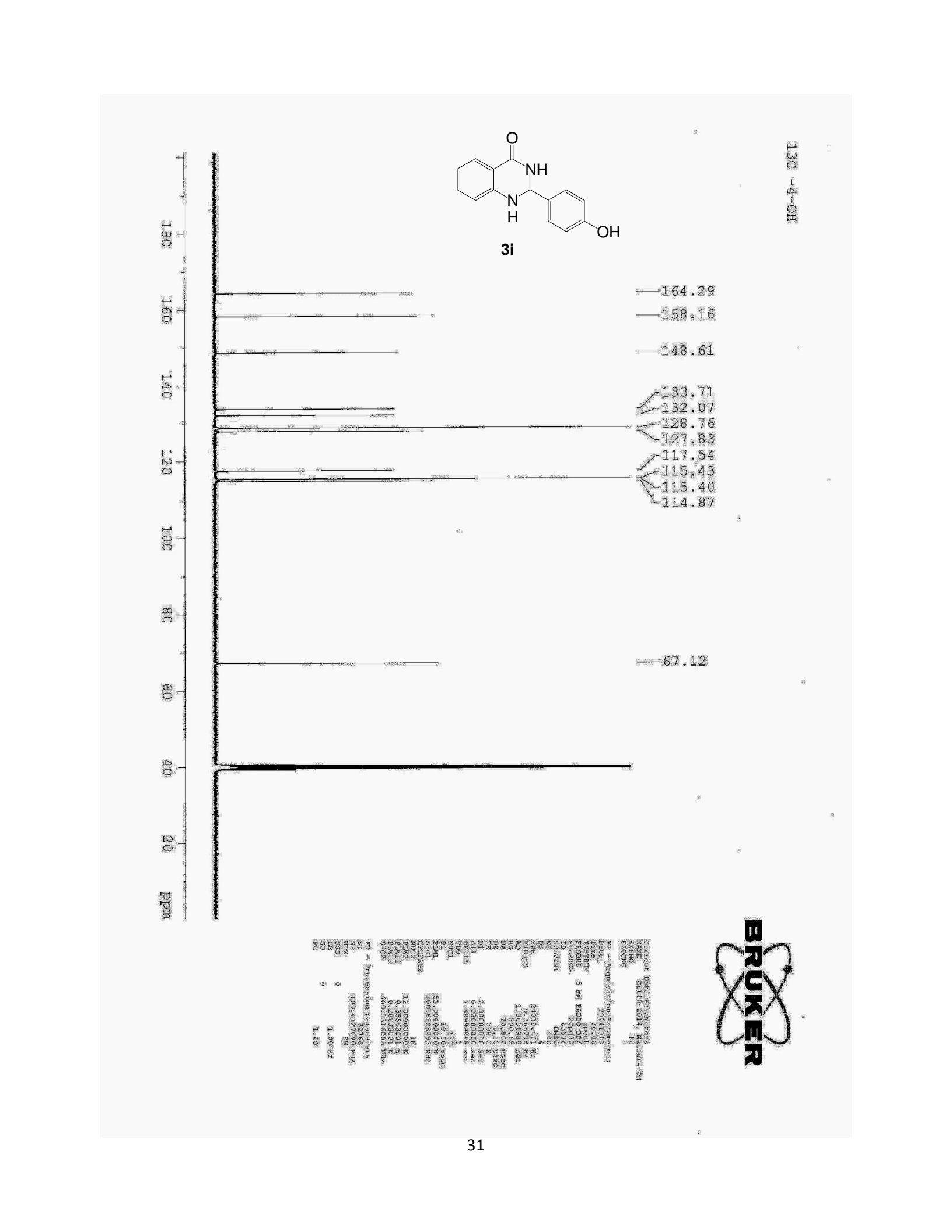

1H-4-Sme-CHO
8.278
7.635
7.616
7.448
7.428
7.296
7.275
7.253
7.236
7.232
7.090
6.768
6.748
6.704
6.686
6.667
5.735
2.470
0.97
0.99
2.11
3.06
0.97
0.99
1.02
1.00
3.02
ppm
O
NH
N
H
SMe
3g
BRUKER


13C-4-Sme-CHO
164.08
148.29
138.92
138.70
133.77
127.91
127.82
126.19
117.62
115.47
114.90
66.58
15.16
ppm
O
NH
N
H
SMe
3g

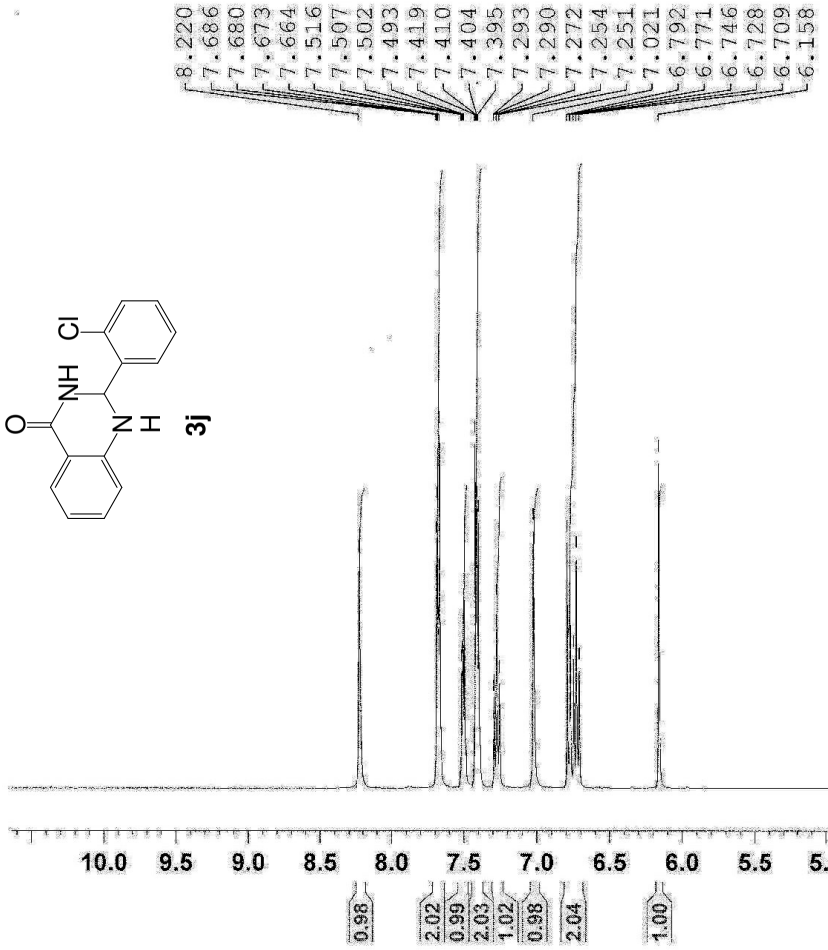

TH--4-CN-CHO
8.484
7.887
7.866
7.687
7.667
7.632
7.612
7.289
7.268
7.251
7.247
6.788
6.768
6.715
6.697
6.678
5.869
0.99
2.00
3.06
2.03
1.00
1.02
1.00
ppm
O
NH
N
H
CN
3h
BRUKER


13C--4-CN-CHO
3h
163.80
147.81
147.79
134.01
132.87
128.15
127.86
119.11
117.90
115.37
114.98
111.54
66.01
40.61
40.40
40.19
39.98
39.77
39.56
39.35
200
180
160
140
120
100
80
60
40
20
0
ppm
BRUKER


1H -4-OH
9.541
8.116
7.648
7.645
7.629
7.626
7.340
7.318
7.269
7.266
7.248
7.231
7.227
6.949
6.801
6.779
6.769
6.748
6.708
6.706
6.689
6.671
6.669
5.678
0.96
1.07
0.96
1.97
1.00
0.99
2.99
1.01
1.00
ppm
3i
Current Data Parameters
NAME
EXPNO
PROCNO
F2 - Acquisition Parameters
Date_
Time
INSTRUM
PROBHD
PULPROG
TD
SOLVENT
NS
DS
SWH
FIDRES
AQ
RG
DW
DE
TE
D1
TD0
======== CHANNEL f1 ========
SFO1
NUC1
P1
PL1
F2 - Processing parameters
SI
SF
WDW
SSB
LB
GB
PC


13C -4-OH
O
NH
N
H
OH
3i
164.29
158.16
148.61
133.71
132.07
128.76
127.83
117.54
115.43
115.40
114.87
67.12
180
160
140
120
100
80
60
40
20
ppm
BRUKER

1H-2-Cl-CHO



Current Data Parameters

NAME	Sep18-2014, Matiur-2-Cl
EXPNO	10
PROCNO	1

F2 - Acquisition Parameters

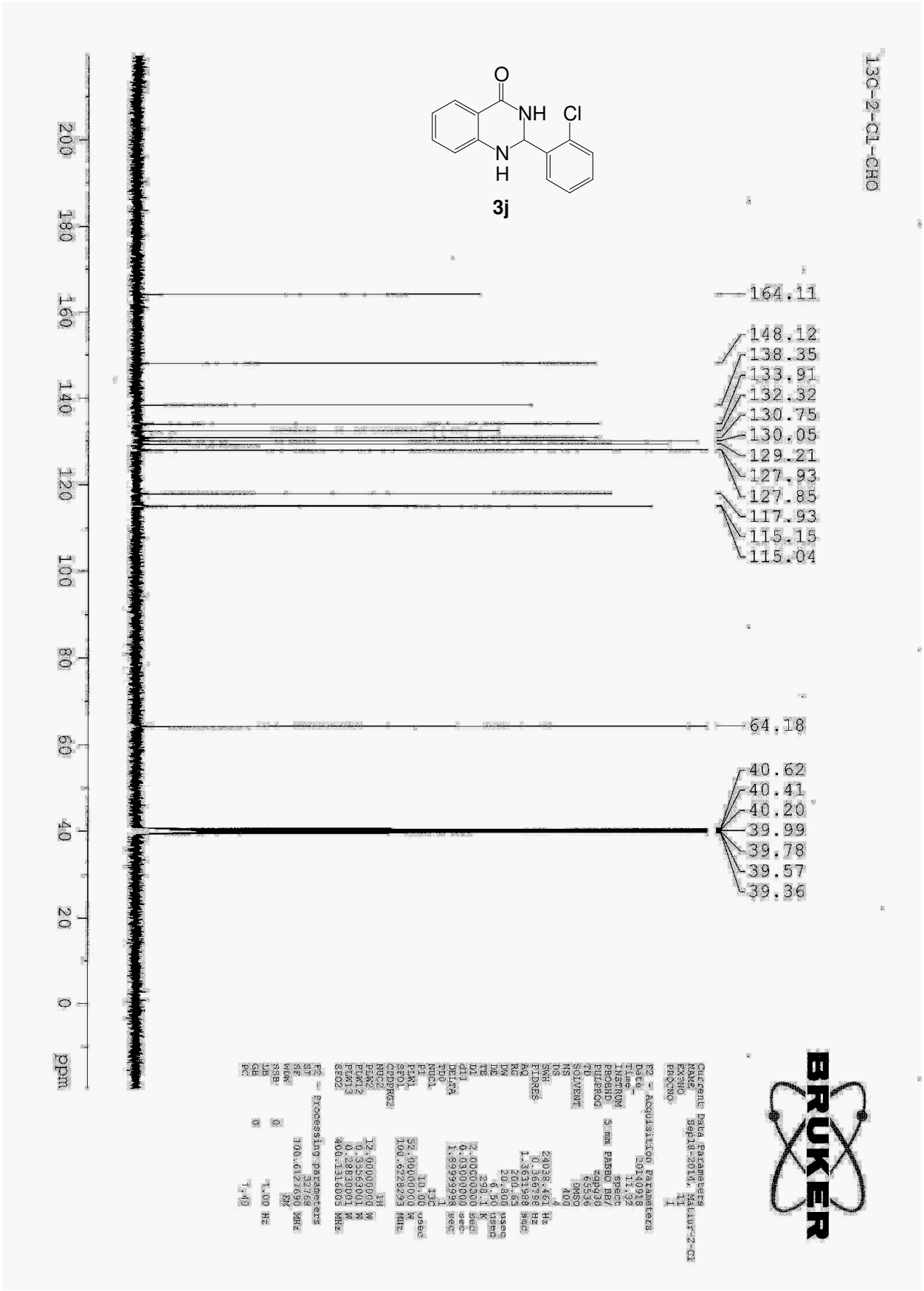
Date_	20140918
Time	11.08
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zg30
TD	65536
SOLVENT	DMSO
NS	16
DS	2
SWH	8012.820 Hz
FIDRES	0.122266 Hz
AQ	4.0894966 sec
RG	99.76
DW	62.400 usec
DE	6.50 usec
TE	298.1 K
D1	1.00000000 sec
TD0	1

======== CHANNEL f1 ========

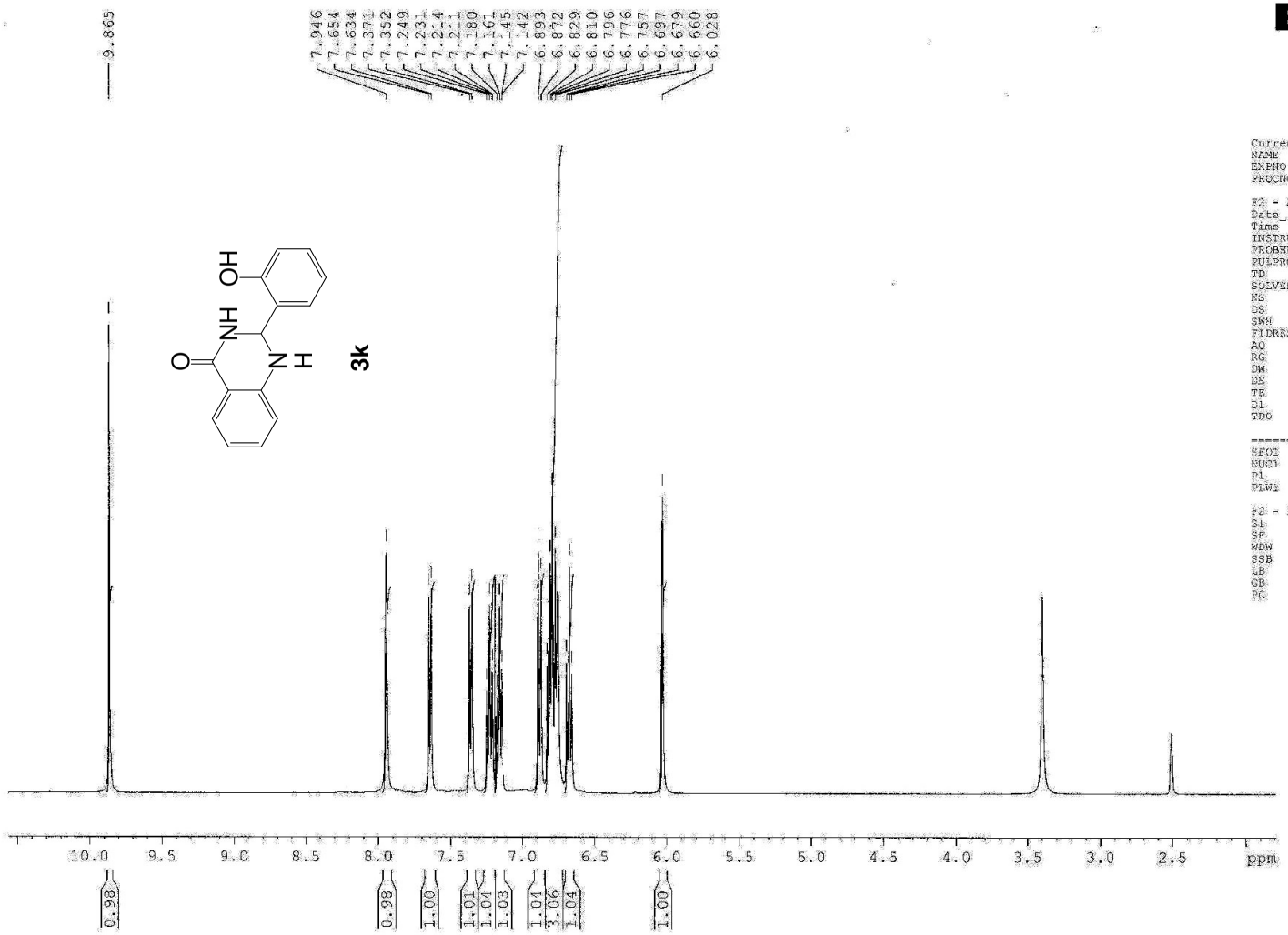
SFO1	400.1324710 MHz
NUC1	1H
P1	15.50 usec
PLW1	12.00000000 W

F2 - Processing parameters

SI	65536
SF	400.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

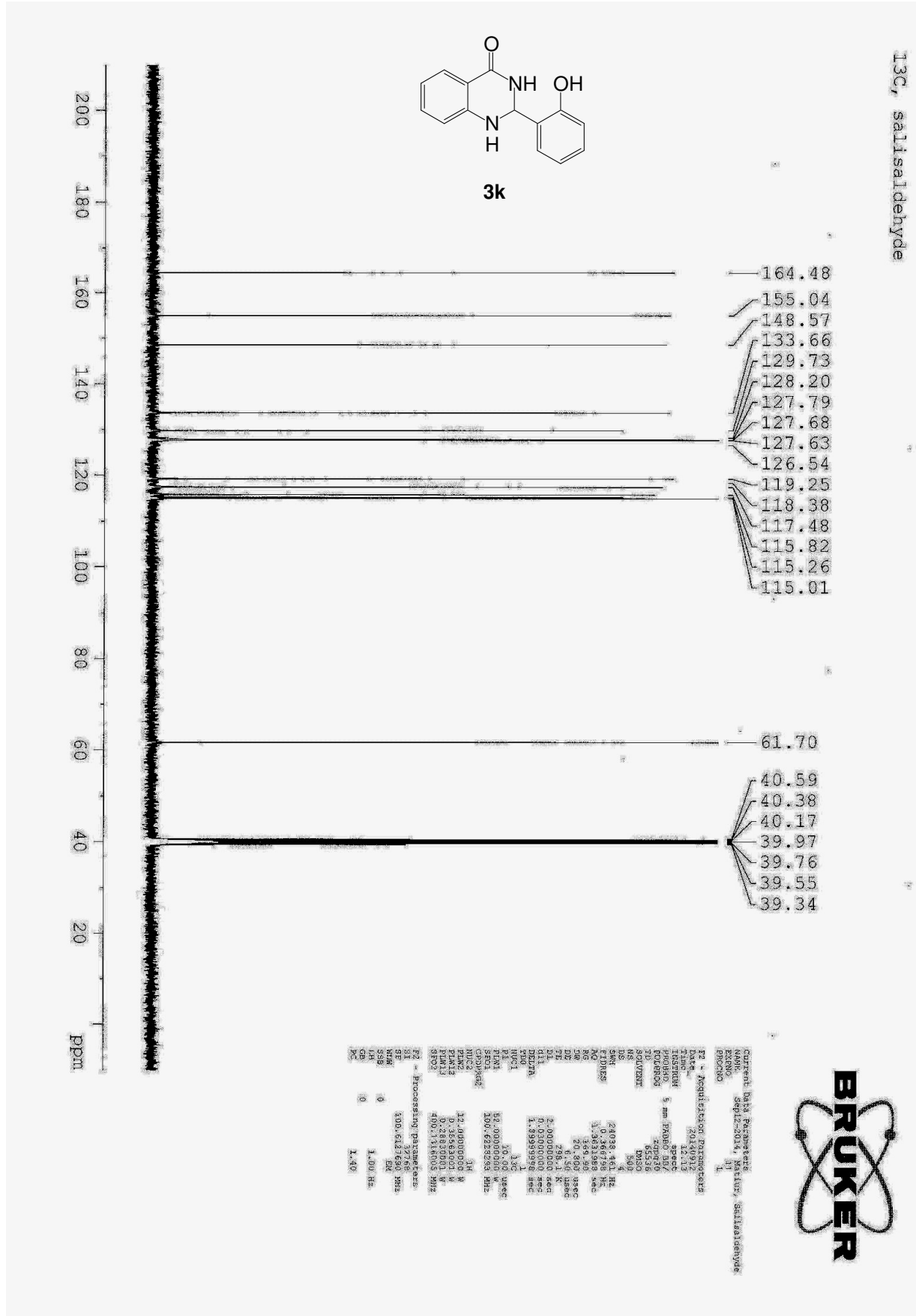

13C-2-Cl-CHO
O
NH
Cl
N
H
3j
164.11
148.12
138.35
133.91
132.32
130.75
130.05
129.21
127.93
127.85
117.93
115.15
115.04
64.18
40.62
40.41
40.20
39.99
39.78
39.57
39.36
200
180
160
140
120
100
80
60
40
20
0
ppm
BRUKER

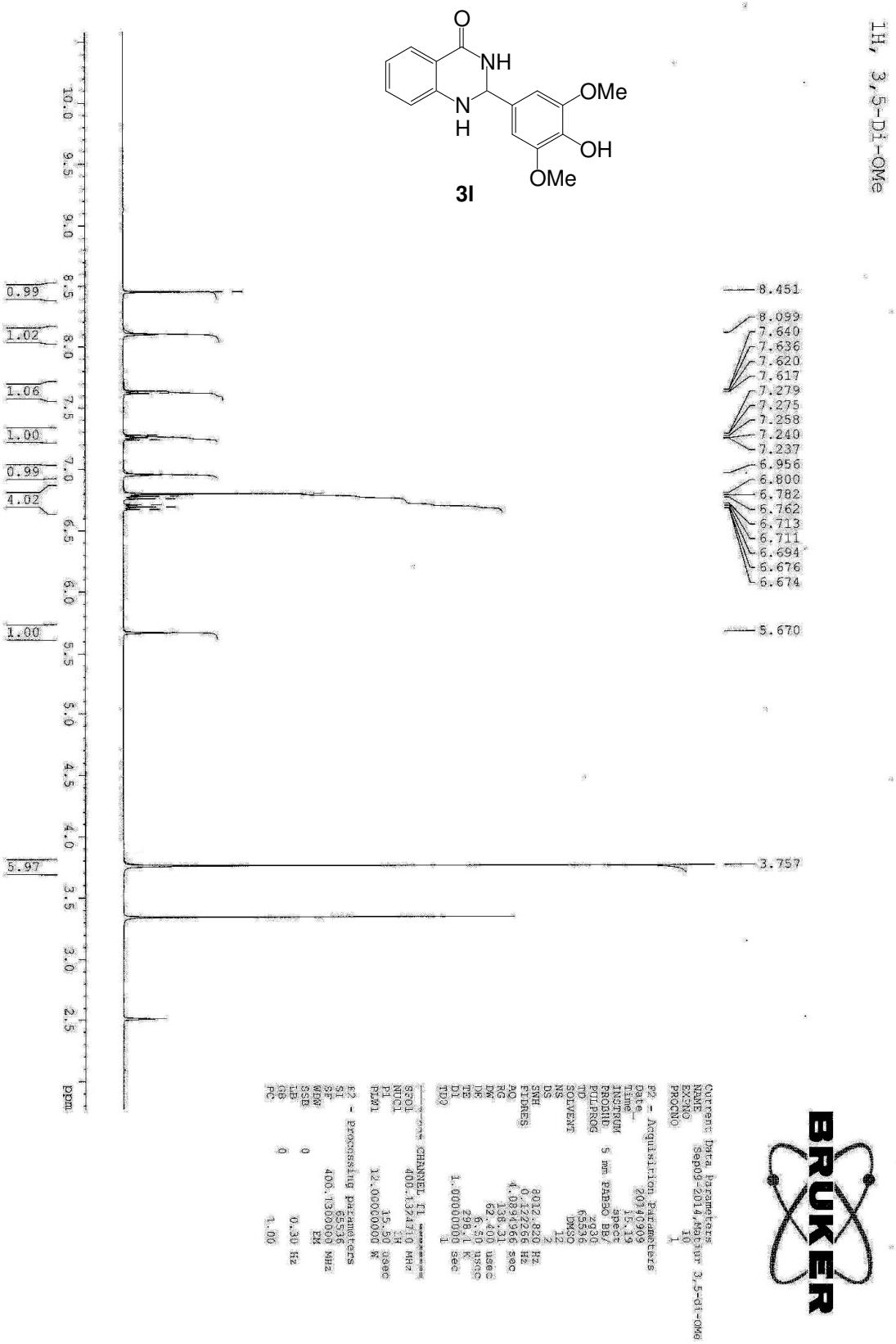
1H, salisaldehyde

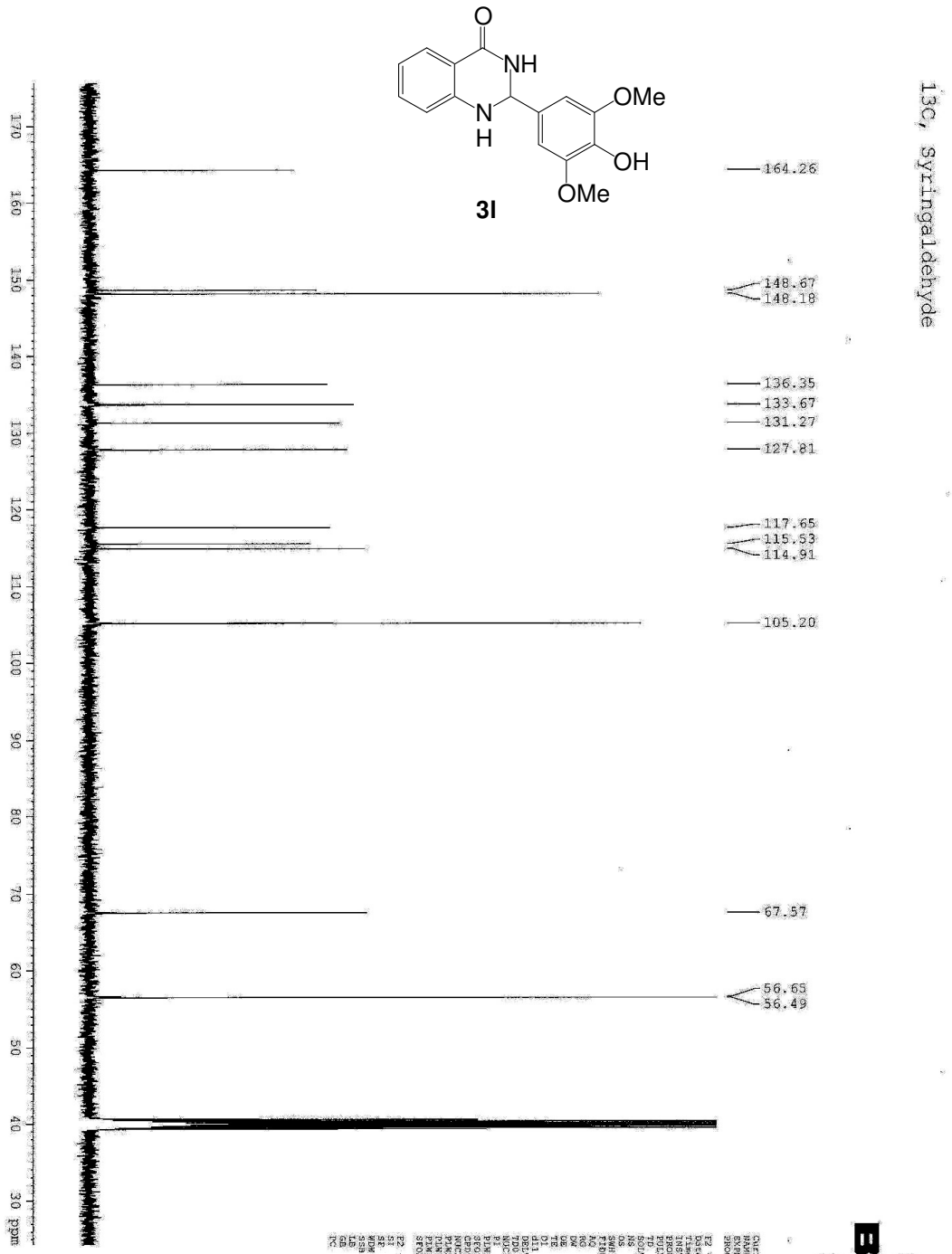


BRUKER

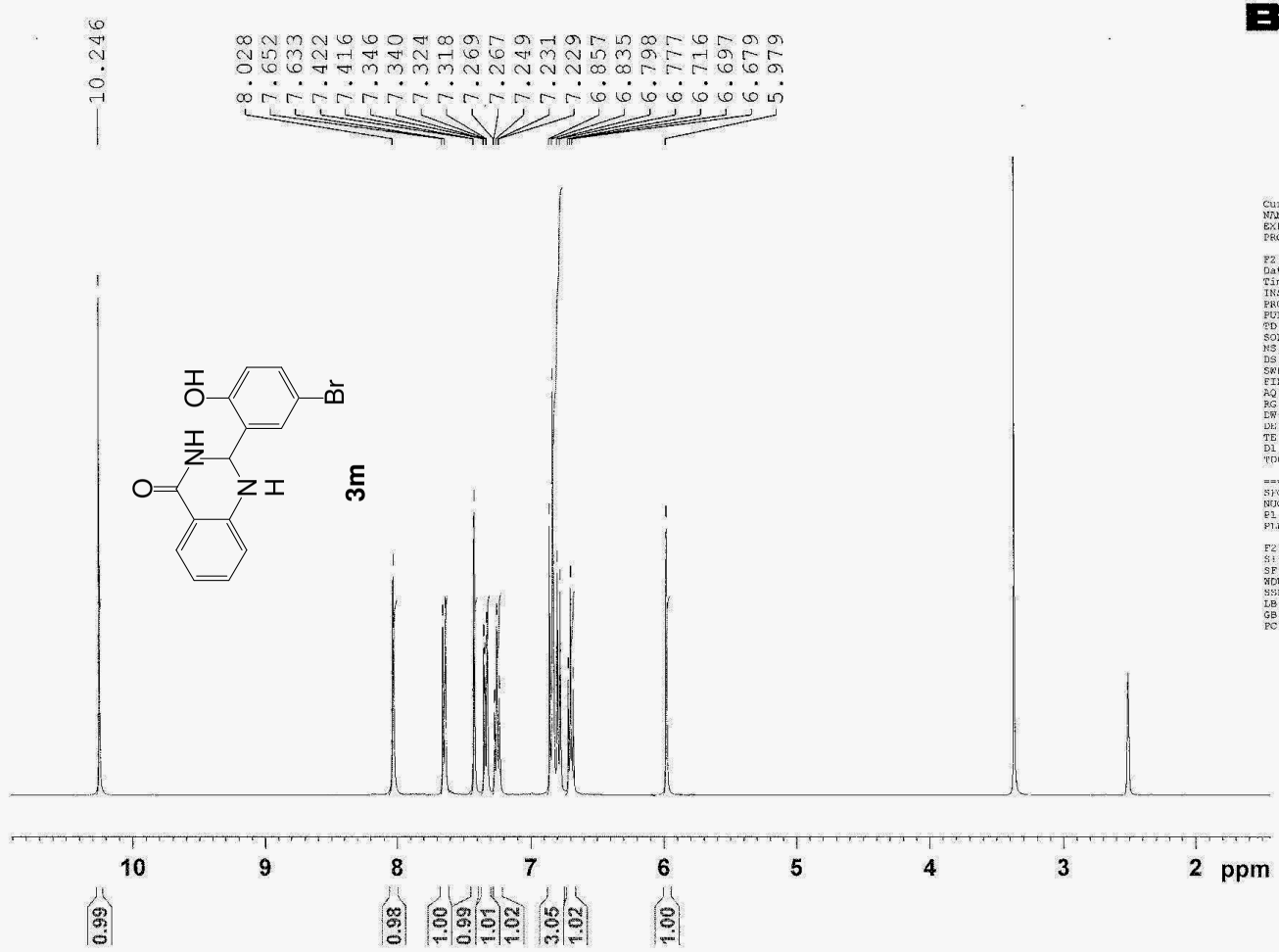
```
Current Data Parameters
NAME      Sep12-2014, Matiur, Salisaldeh
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140912
Time              11.43
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG           zg30
TD               65536
SOLVENT           DMSO
NS                   12
DS                    2
SWH           8012.820 Hz
FIDRES        0.122266 Hz
AQ            4.0894966 sec
RG                88.03
DW               62.400 usec
DE                6.50 usec
TE              298.2 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        400.1324710 MHz
NUC1                1H
P1                15.50 usec
PLW1        12.00000000 W

F2 - Processing parameters
SI                65536
SF          400.1300000 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```


13C, salisaldehyde
O
NH
OH
N
H
3k
164.48
155.04
148.57
133.66
129.73
128.20
127.79
127.68
127.63
126.54
119.25
118.38
117.48
115.82
115.26
115.01
61.70
40.59
40.38
40.17
39.97
39.76
39.55
39.34
200
180
160
140
120
100
80
60
40
20
ppm
BRUKER


1H, 3,5-Di-OMe
O
NH
N
H
OMe
OH
OMe
3l
8.451
8.099
7.640
7.636
7.620
7.617
7.279
7.275
7.258
7.240
7.237
6.956
6.800
6.782
6.762
6.713
6.711
6.694
6.676
6.674
5.670
3.757
0.99
1.02
1.06
1.00
0.99
4.02
1.00
5.97
ppm
Current Data Parameters
NAME Sep09-2014/Mohur 3,5-di-OMe
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140909
Time 15.19
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 12
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 138.31
DW 62.400 usec
DE 6.50 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 15.50 usec
PLW1 12.00000000 W
F2 - Processing parameters
SI 65536
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

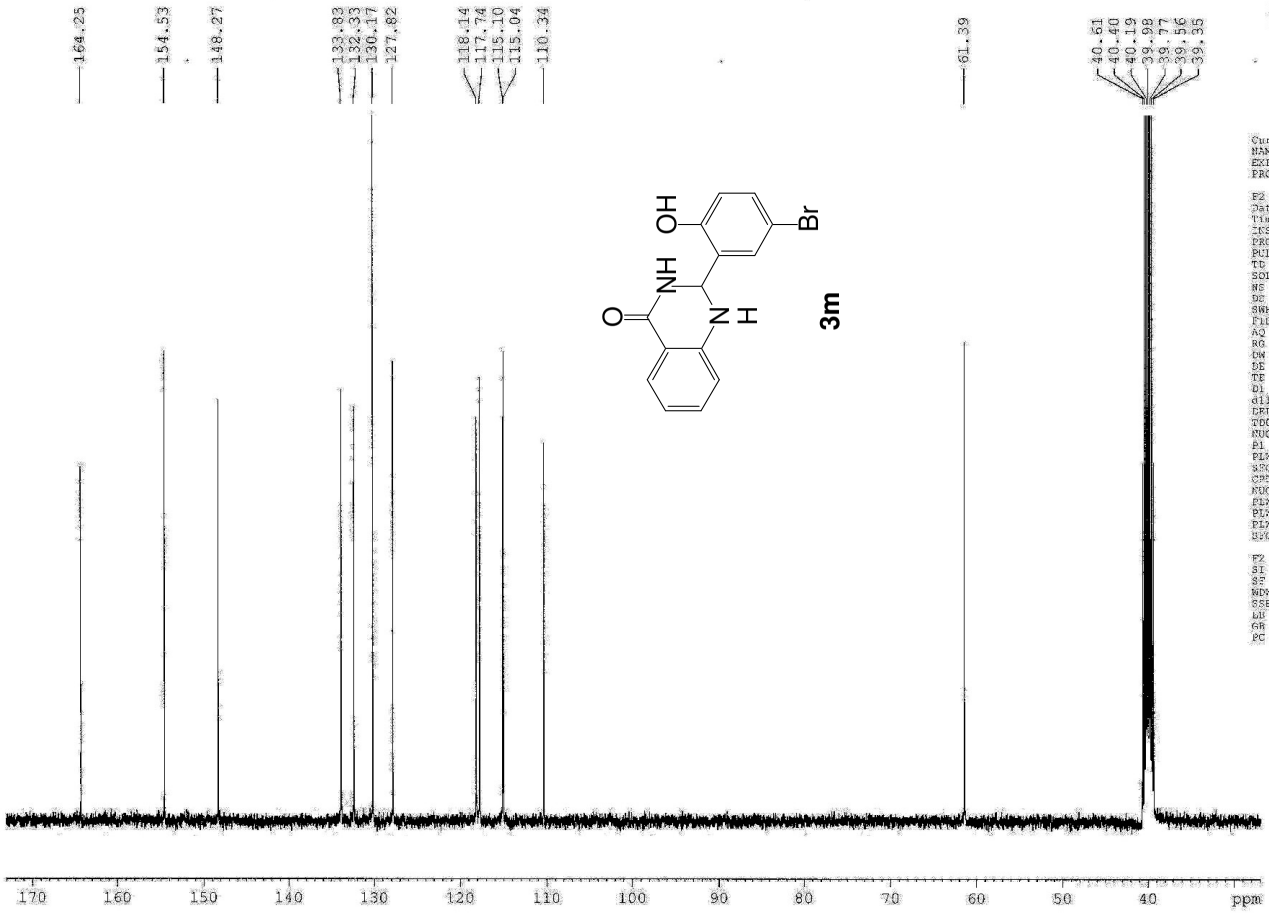

13C, Syringaldehyde
O
NH
N
H
OMe
OH
OMe
3l
164.26
148.67
148.18
136.35
133.67
131.27
127.81
117.65
115.53
114.91
105.20
67.57
56.65
56.49
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
ppm
BRUKER

1H-3-Br-sali



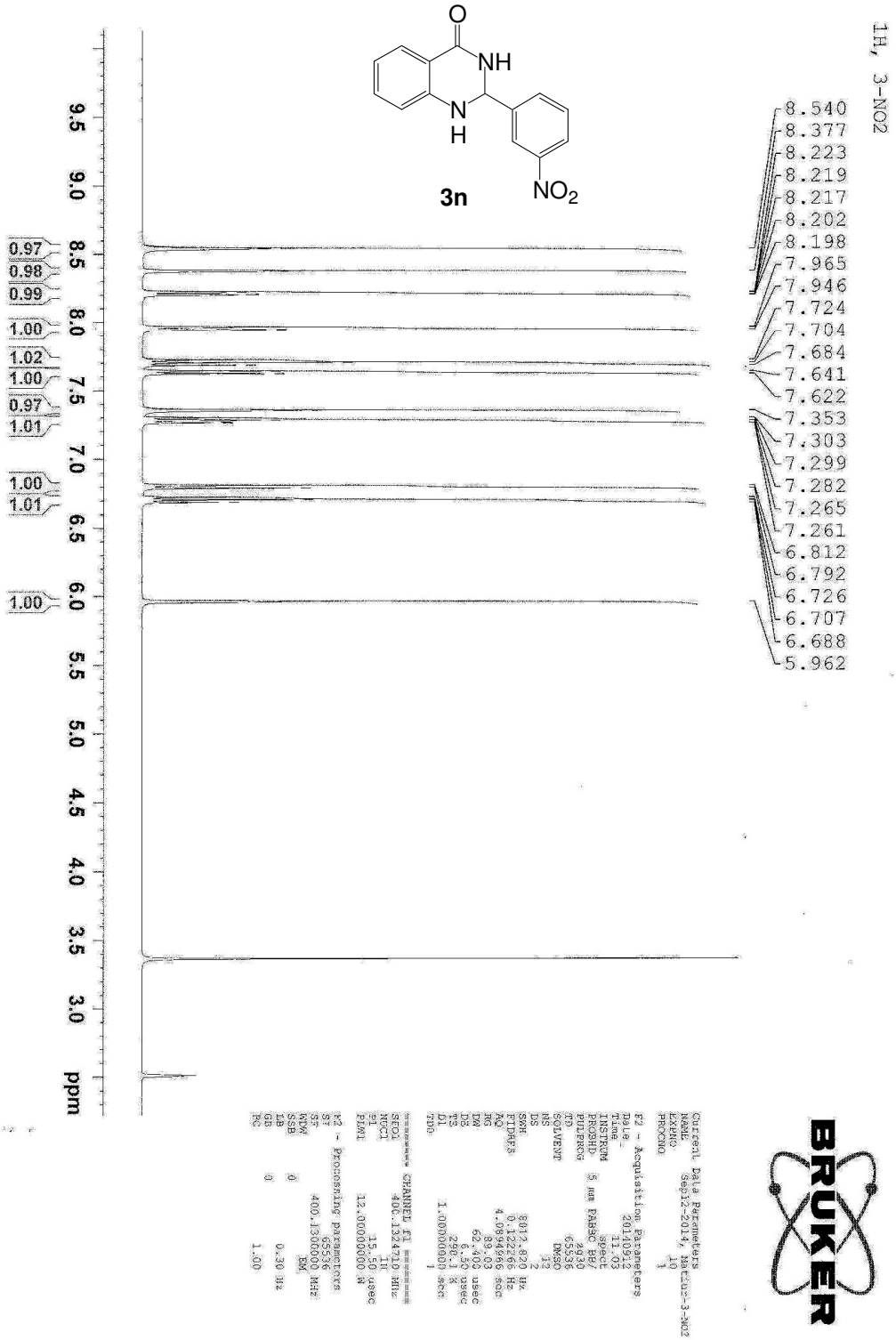

```
Current Data Parameters
NAME       Sep22-2014, Matiur-3-Br-sali
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140922
Time              12.29
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG           zg30
TD               65536
SOLVENT           DMSO
NS                   32
DS                    2
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894966 sec
RG                123.93
DW                62.400 usec
DE                 6.50 usec
TE               298.1 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1               1H
P1                15.50 usec
PLW1        12.00000000 W

F2 - Processing parameters
SI                65536
SF           400.1300000 MHz
WDW                  EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

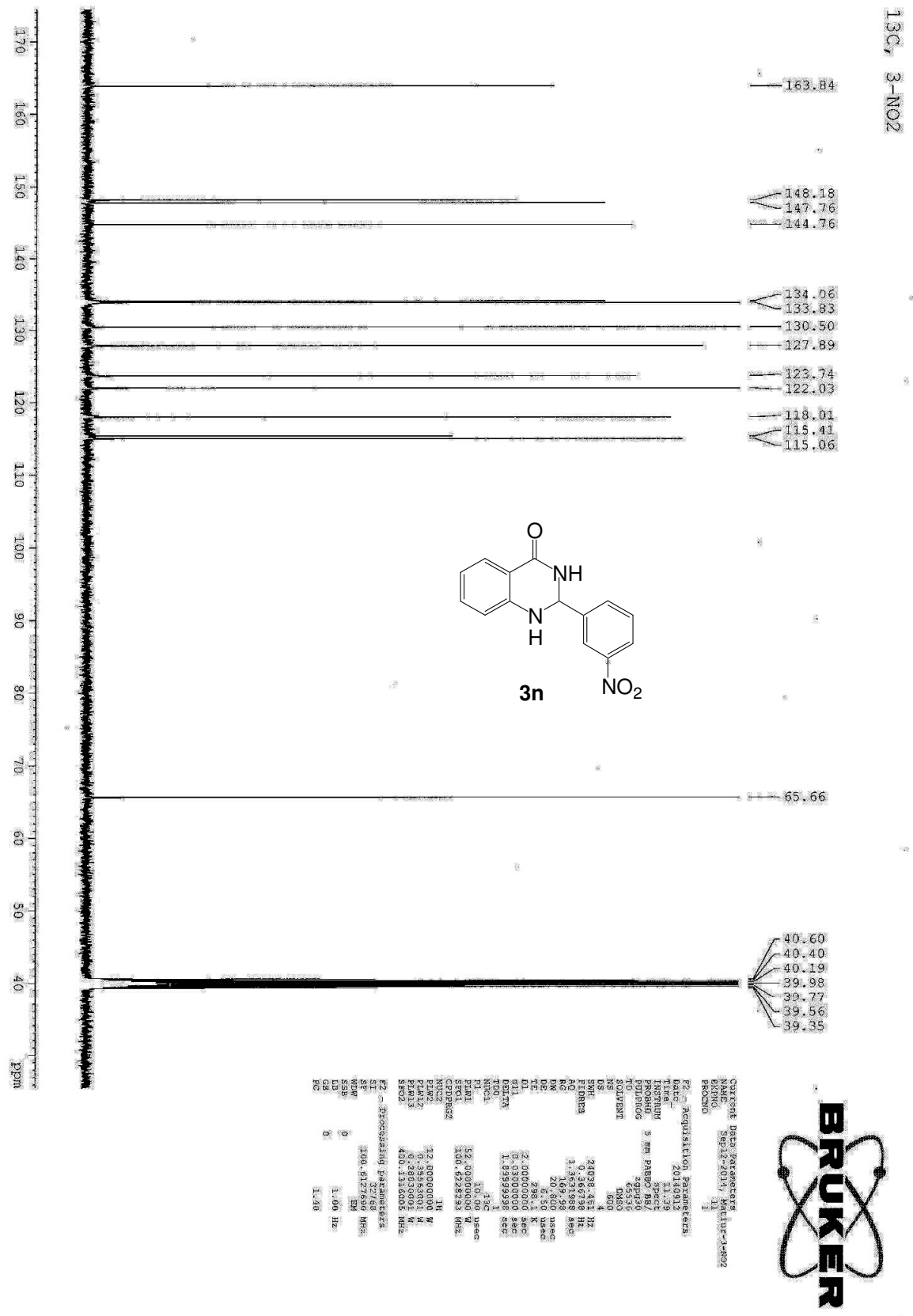
13C-3-Br-sali



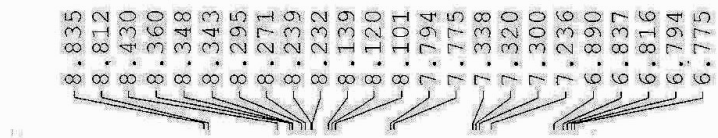
BRUKER

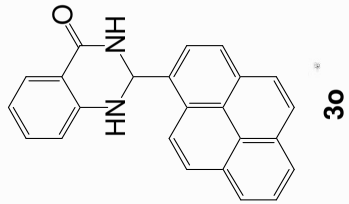
```
Current Data Parameters
NAME      Sep22-2014, Mathur-3-Br-sali
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140922
Time              13.05
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT            DMSO
NS                   600
DS                     4
SWH           24038.461 Hz
FIDRES        0.366799 Hz
AQ            1.3631988 sec
RG                206.65
DW               20.800 usec
DE                6.50 usec
TE              298.1 K
D1              2.00000000 sec
d11             0.03000000 sec
DELTA           1.89999998 sec
TD0                    1
NUC1                 13C
P1                 10.00 usec
PLW1        52.00000000 W
SFO1          100.6228293 MHz
CPDPRG2
NUC2                 1H
PLW2        12.00000000 W
PLW12        0.35563001 W
PLW13        0.28830001 W
SFO2          400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF            100.6127690 MHz
WDW                  EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40
```


1H, 3-NO2
8.540
8.377
8.223
8.219
8.217
8.202
8.198
7.965
7.946
7.724
7.704
7.684
7.641
7.622
7.353
7.303
7.299
7.282
7.265
7.261
6.812
6.792
6.726
6.707
6.688
5.962
O
NH
N
H
NO2
3n
0.97
0.98
0.99
1.00
1.02
1.00
0.97
1.01
1.00
1.01
1.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
ppm
Current Data Parameters
NAME Sep12-2014, Matrix-3-NO2
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140912
Time 11.03
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 32
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894066 sec
RG 89.03
DW 62.400 usec
DE 6.50 usec
TE 290.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 15.50 usec
PLW1 12.00000000 W
F2 - Processing parameters
SI 65536
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


13C, 3-NO2
163.84
148.18
147.76
144.76
134.06
133.83
130.50
127.89
123.74
122.03
118.01
115.41
115.06
65.66
40.60
40.40
40.19
39.98
39.77
39.56
39.35
ppm
O
NH
N
H
NO2
3n
BRUKER

1H-Pyrene-CHO

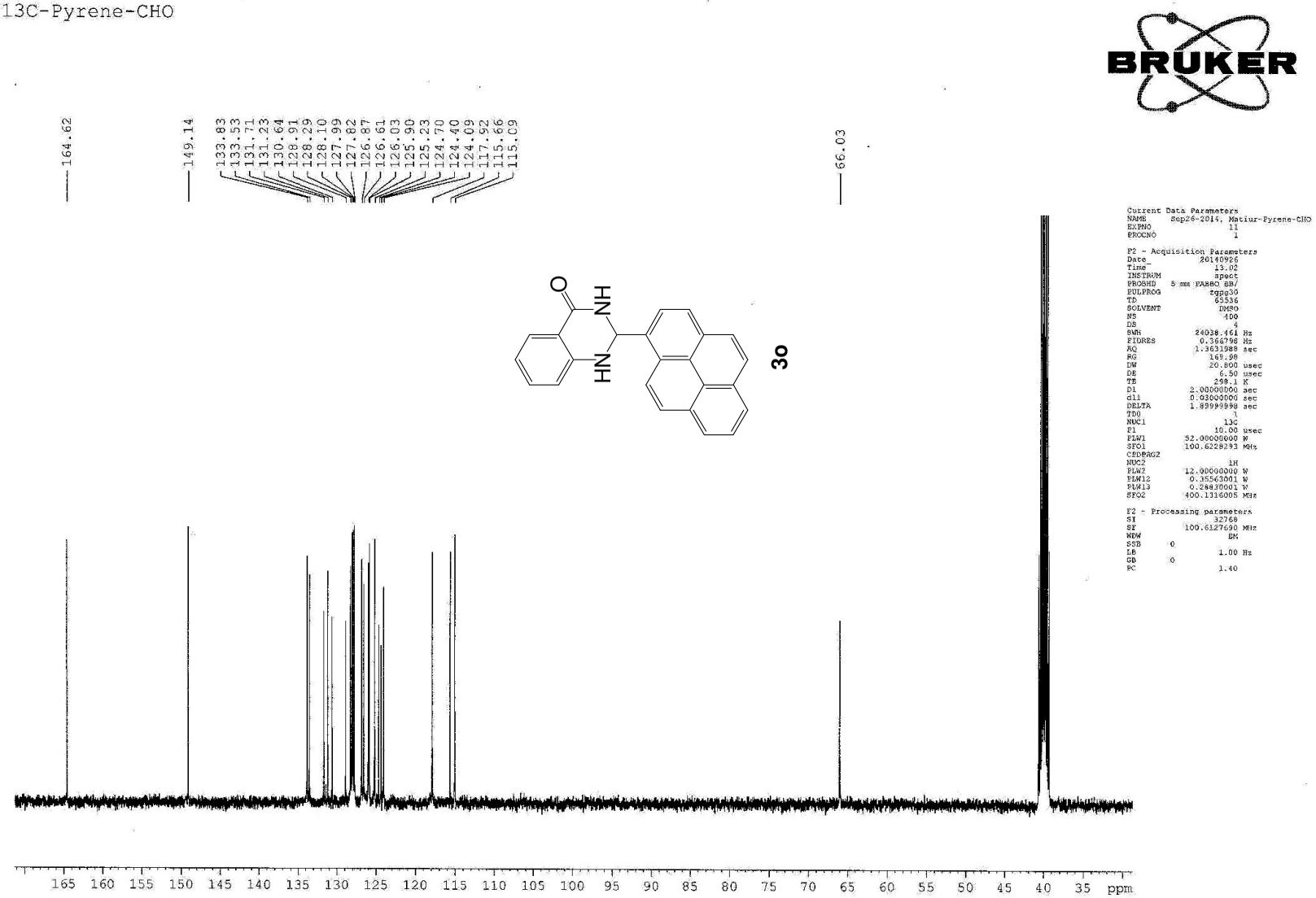




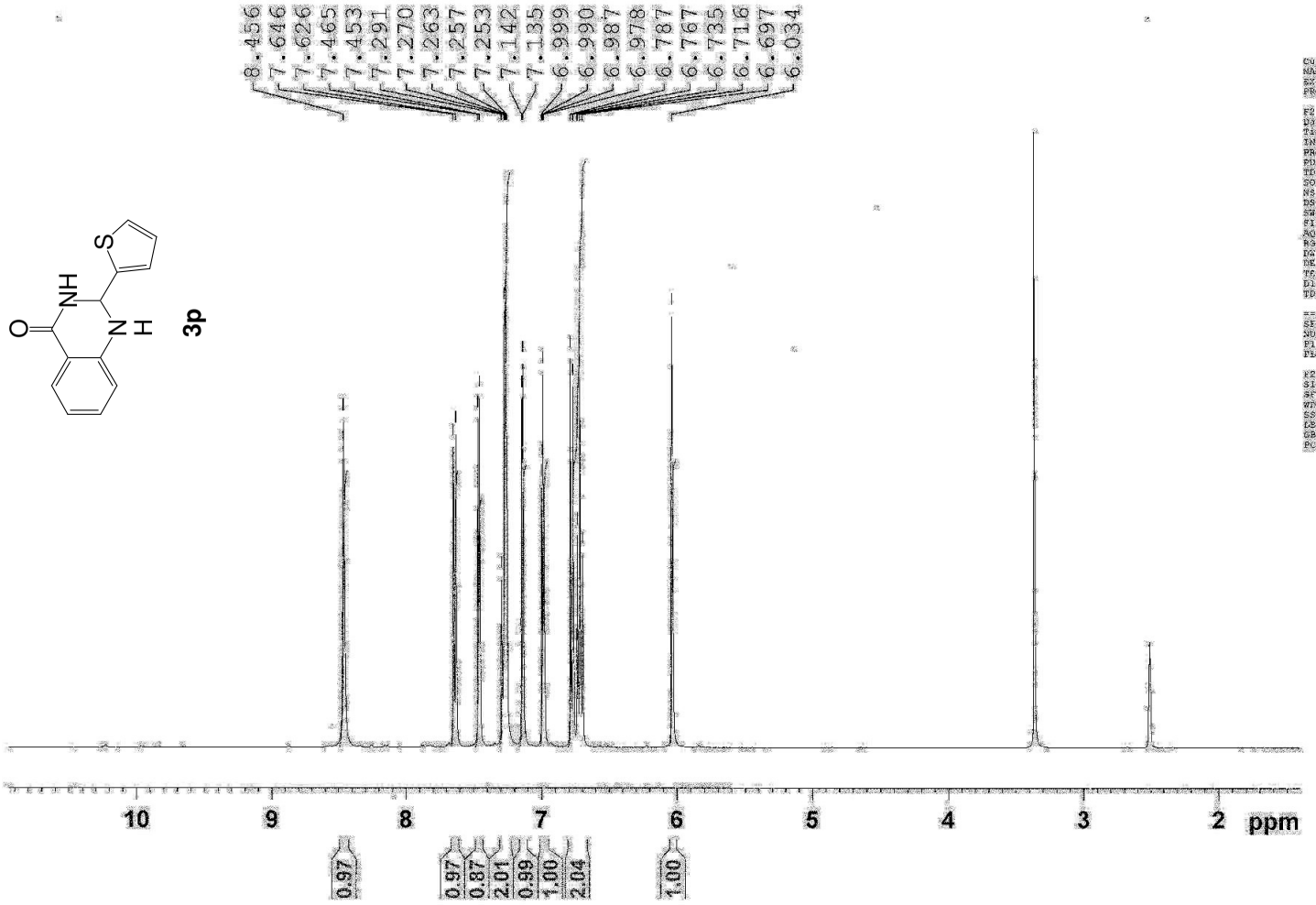
30


```
Current Data Parameters
NAME      Sep25-2014, Matlur-Pyrene-CHO
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140925
Time_              11.20
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT             DMSO
NS                    32
DS                     2
SWH              8012.820 Hz
FIDRES           0.122266 Hz
AQ               4.0894966 sec
RG                 107.78
DW                62.400 usec
DE                 6.50 usec
TE                298.1 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                  15.50 usec
PLW1         12.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.1300000 MHz
WDW                   EM
SSB          0
LB                  0.30 Hz
GB           0
PC                  1.00
```

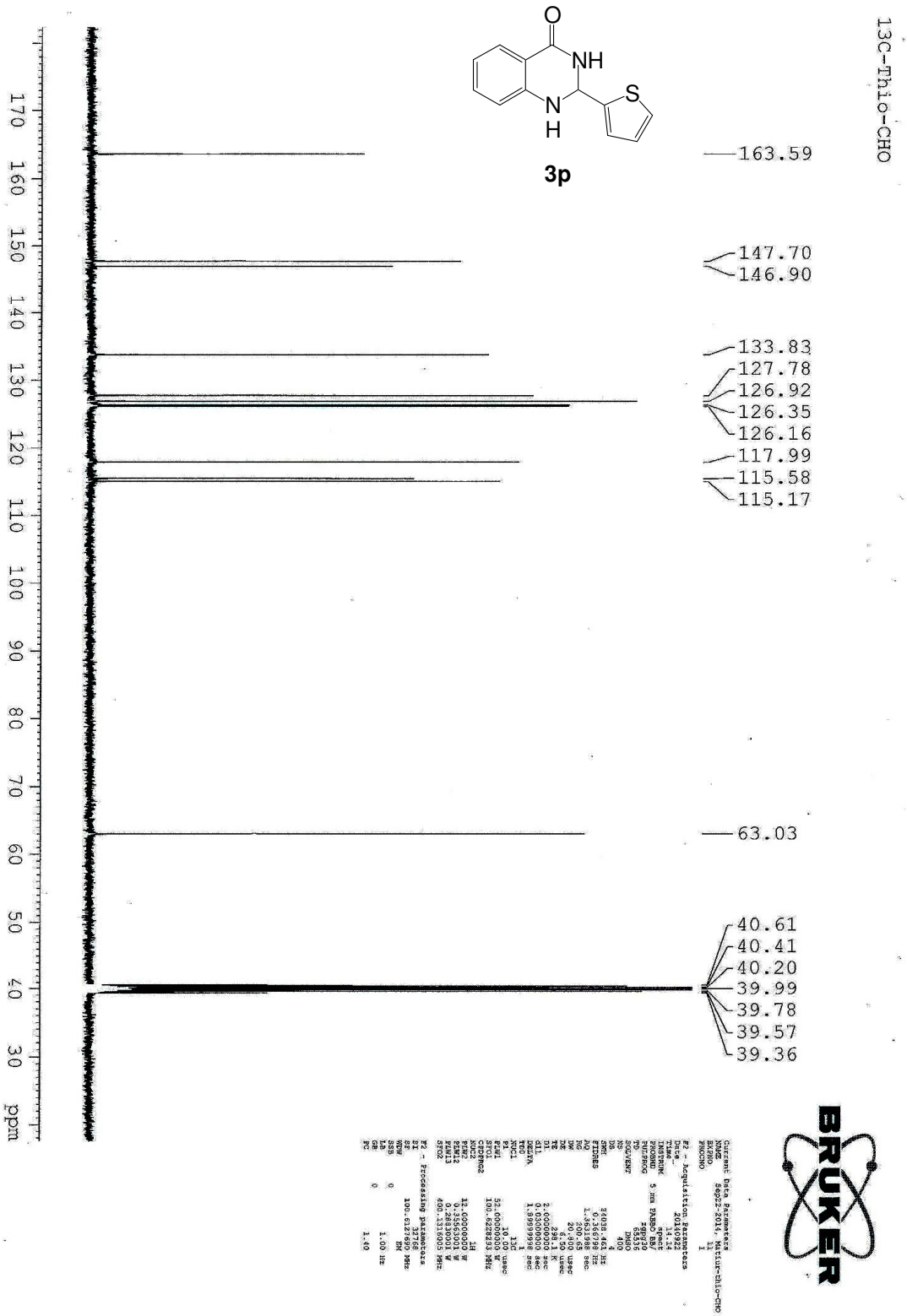
13C-Pyrene-CHO



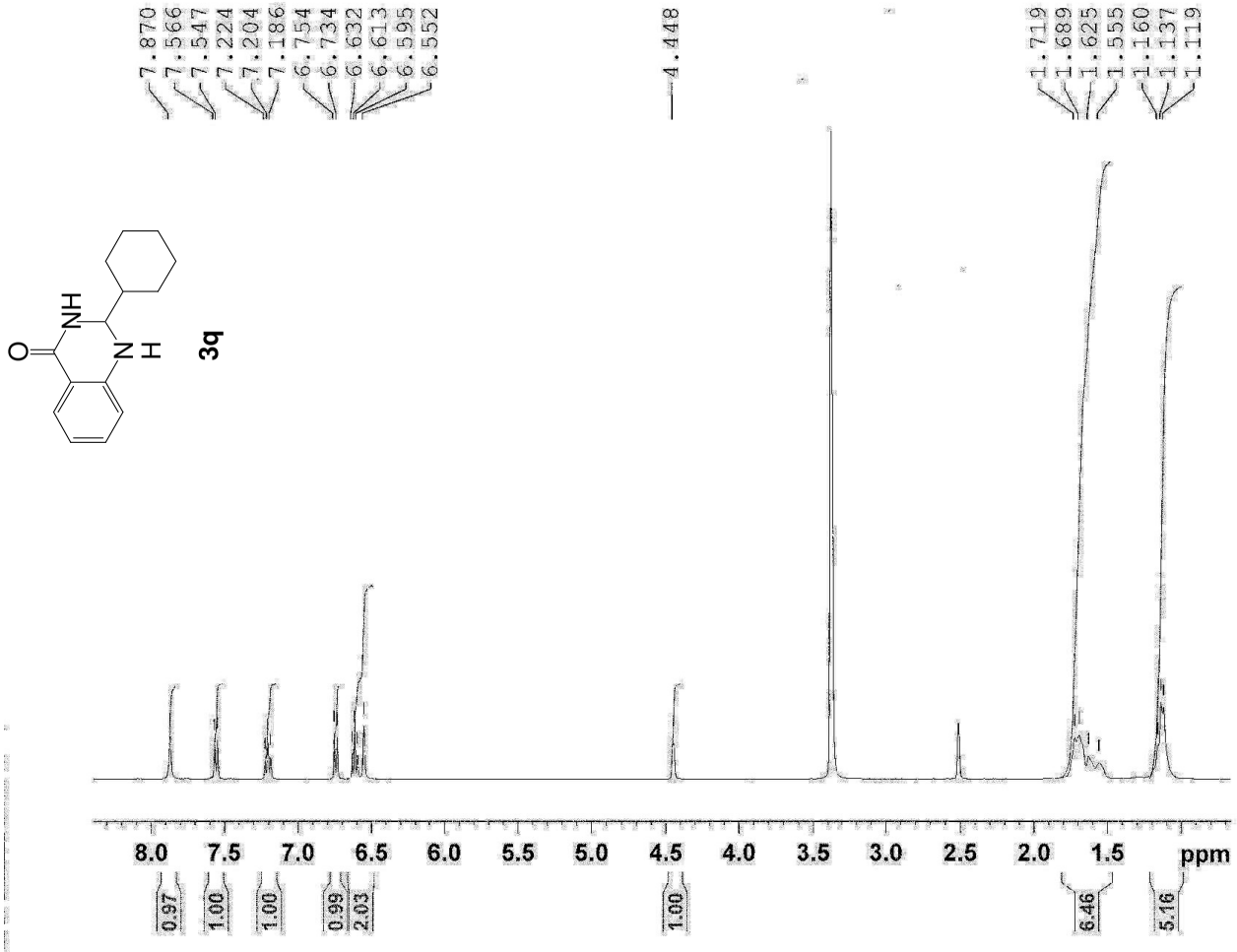
1H-Thio-CHO




```
Current Data Parameters
NAME        Sep22-2014, Mathur-thio-CHO
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_          20140922
Time              13.50
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG           zg30
TD                65536
SOLVENT             DMSO
NS                    16
DS                     2
SWH            8013.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894966 sec
RG                 39.96
DW                62.400 usec
DE                 6.50 usec
TE                298.1 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                 15.50 usec
PLW1          12.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.1300000 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

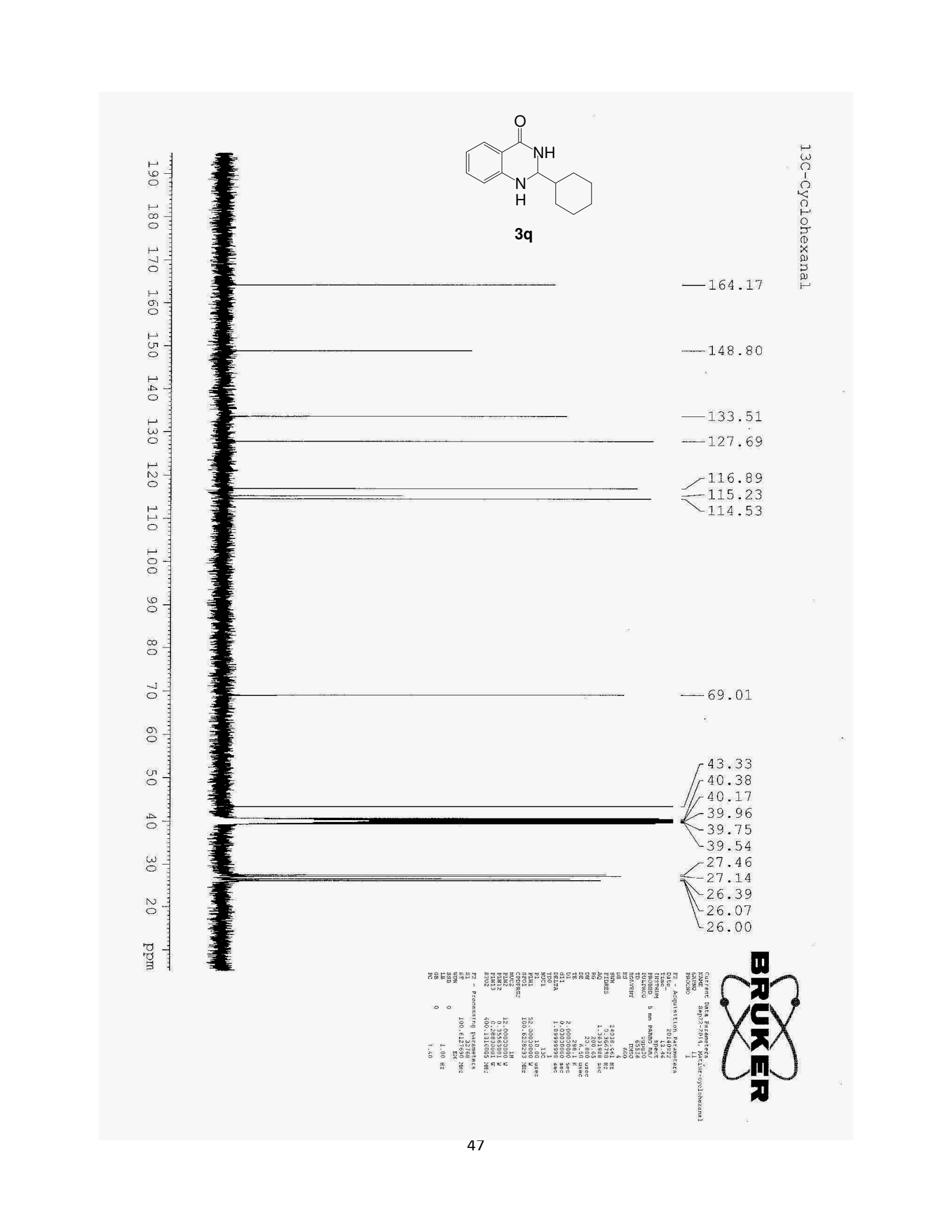

13C-Thio-CHO
O
NH
N
H
S
3p
163.59
147.70
146.90
133.83
127.78
126.92
126.35
126.16
117.99
115.58
115.17
63.03
40.61
40.41
40.20
39.99
39.78
39.57
39.36
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 ppm
BRUKER

1H-Cyclohexanal

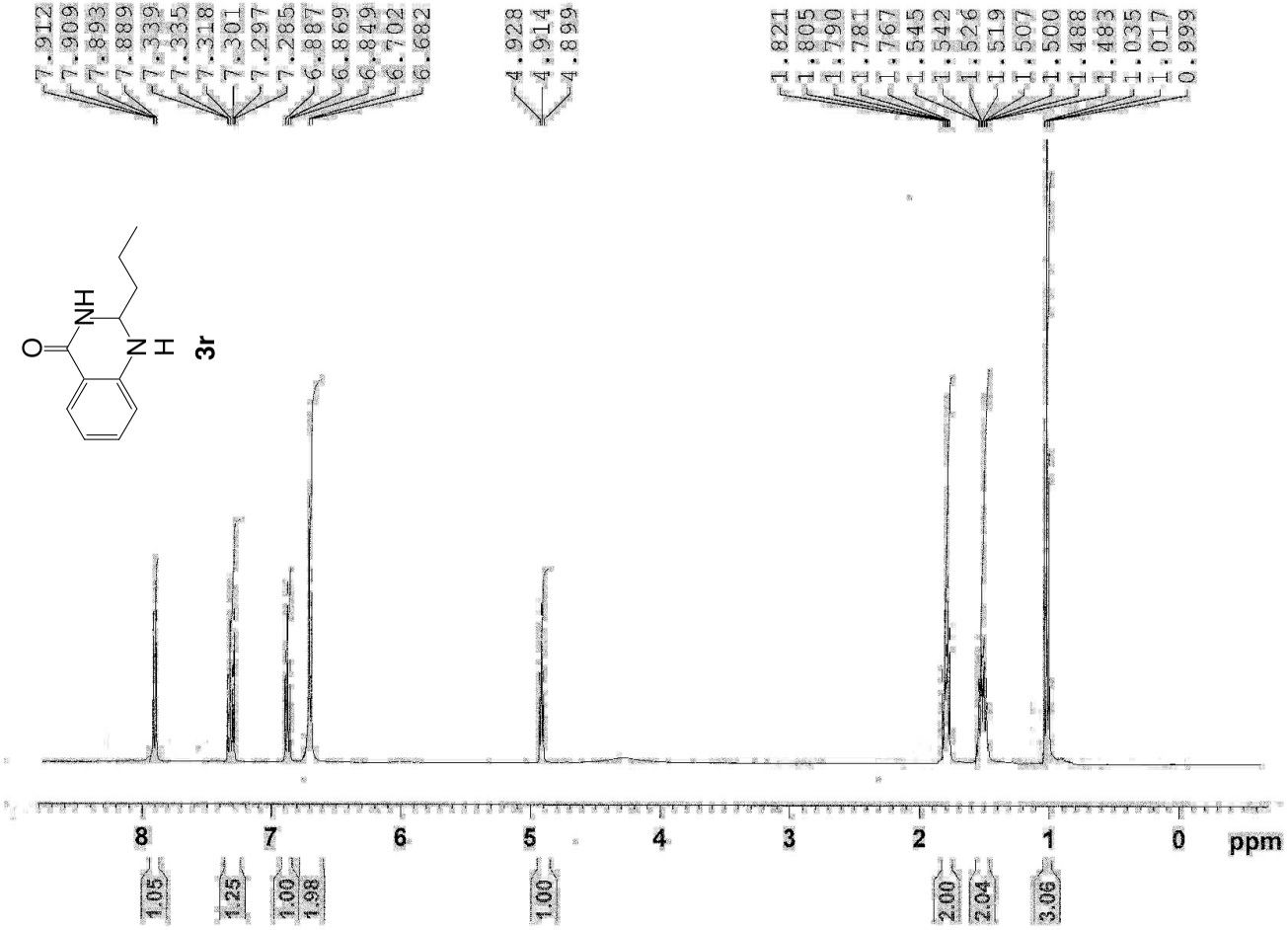


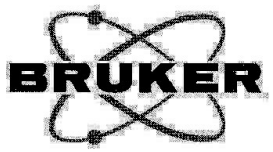
BRUKER

```
Current Data Parameters
NAME        Sep22-2014, Matur-cyclohexanal
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_          20140922
Time           13.10
INSTRUM             spect
PROBHD     5 mm PABBO BB/
PULPROG          zg30
TD              65536
SOLVENT           DMSO
NS                   32
DS                    2
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0899366 sec
RG                99.76
DW               62.400 usec
DE                6.50 usec
TE              298.1 K
D1              1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1          400.1324710 MHz
NUC1                  1H
P1                15.50 usec
PLW1          12.00000000 W

F2 - Processing parameters
SI                65536
SF          400.1300000 MHz
WDW                   EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

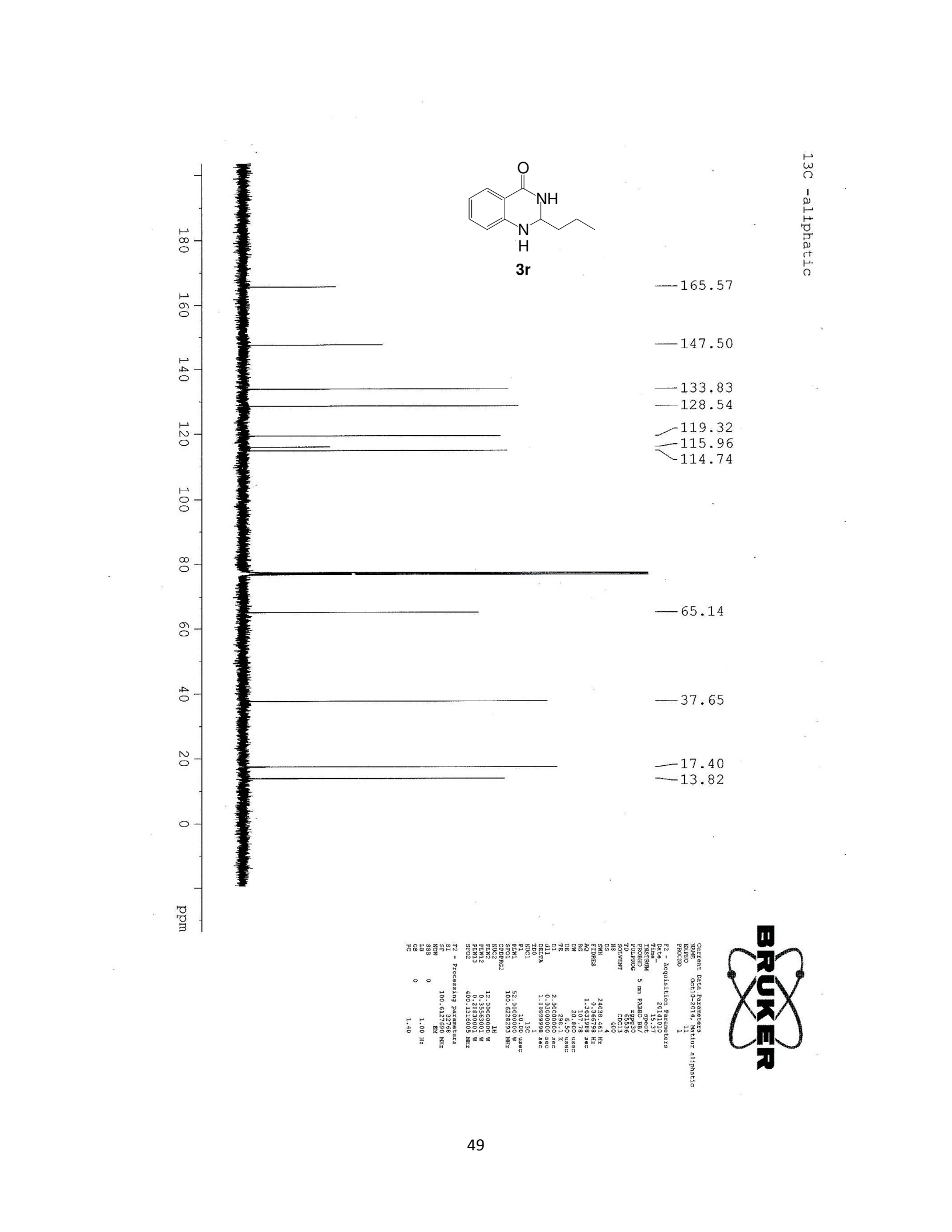

13C-Cyclohexanal
O
NH
N
H
3q
164.17
148.80
133.51
127.69
116.89
115.23
114.53
69.01
43.33
40.38
40.17
39.96
39.75
39.54
27.46
27.14
26.39
26.07
26.00
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm
BRUKER

1H -aliphatic

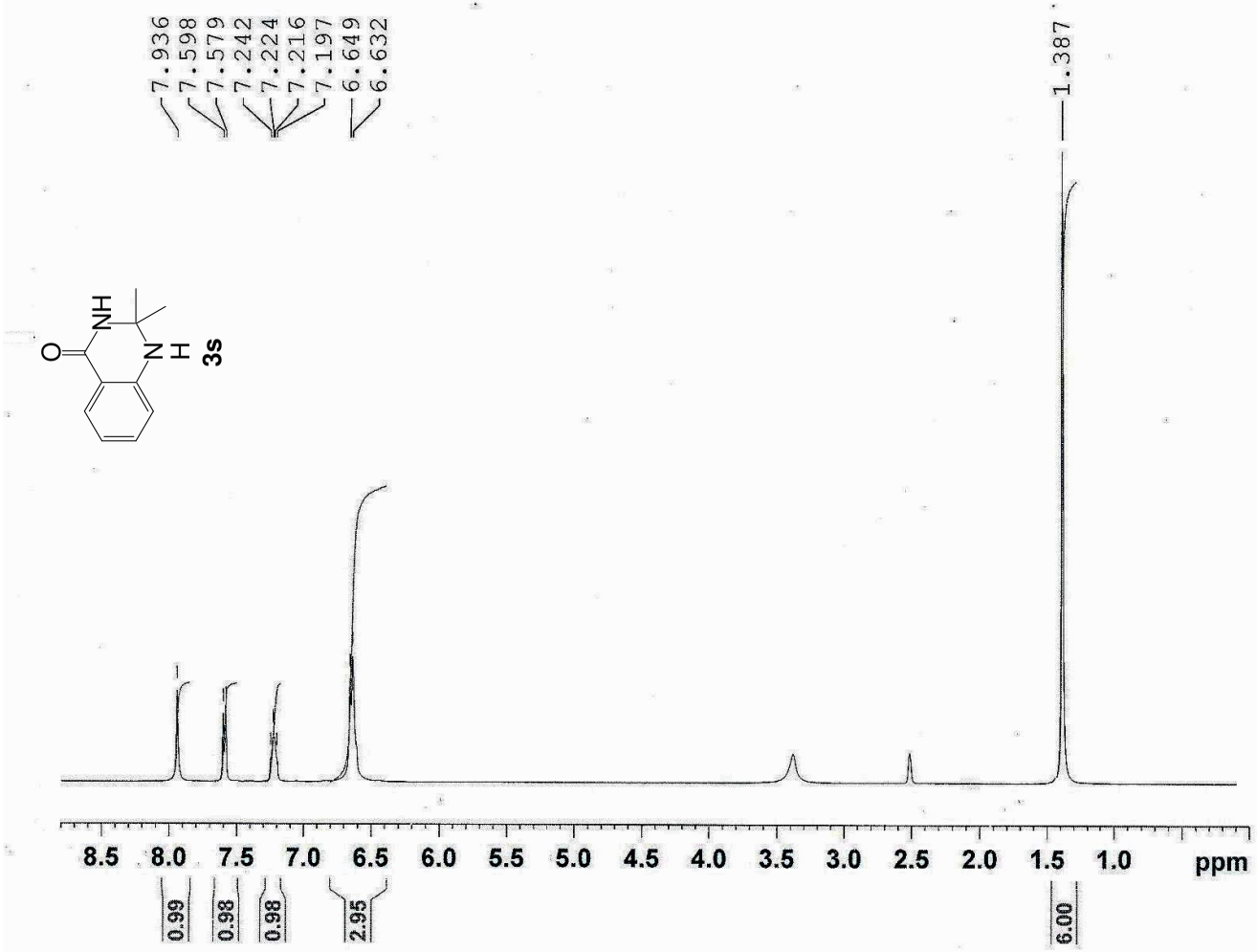





```
Current Data Parameters
NAME       Oct10-2014, Matlur aliphatic
EXPNO              10
PROCNO              1

F2 - Acquisition Parameters
Date_          20141010
Time               15.13
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           8012.820 Hz
FIDRES        0.122266 Hz
AQ            4.0894966 sec
RG                138.31
DW               62.400 usec
DE                6.50 usec
TE               298.1 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         400.1324719 MHz
NUC1               1H
P1                15.50 usec
PLW1         12.00000000 W

F2 - Processing parameters
SI                65536
SF           400.1300000 MHz
WDW                EM
SSB          0
LB                0.30 Hz
GB           0
PC                1.00
```

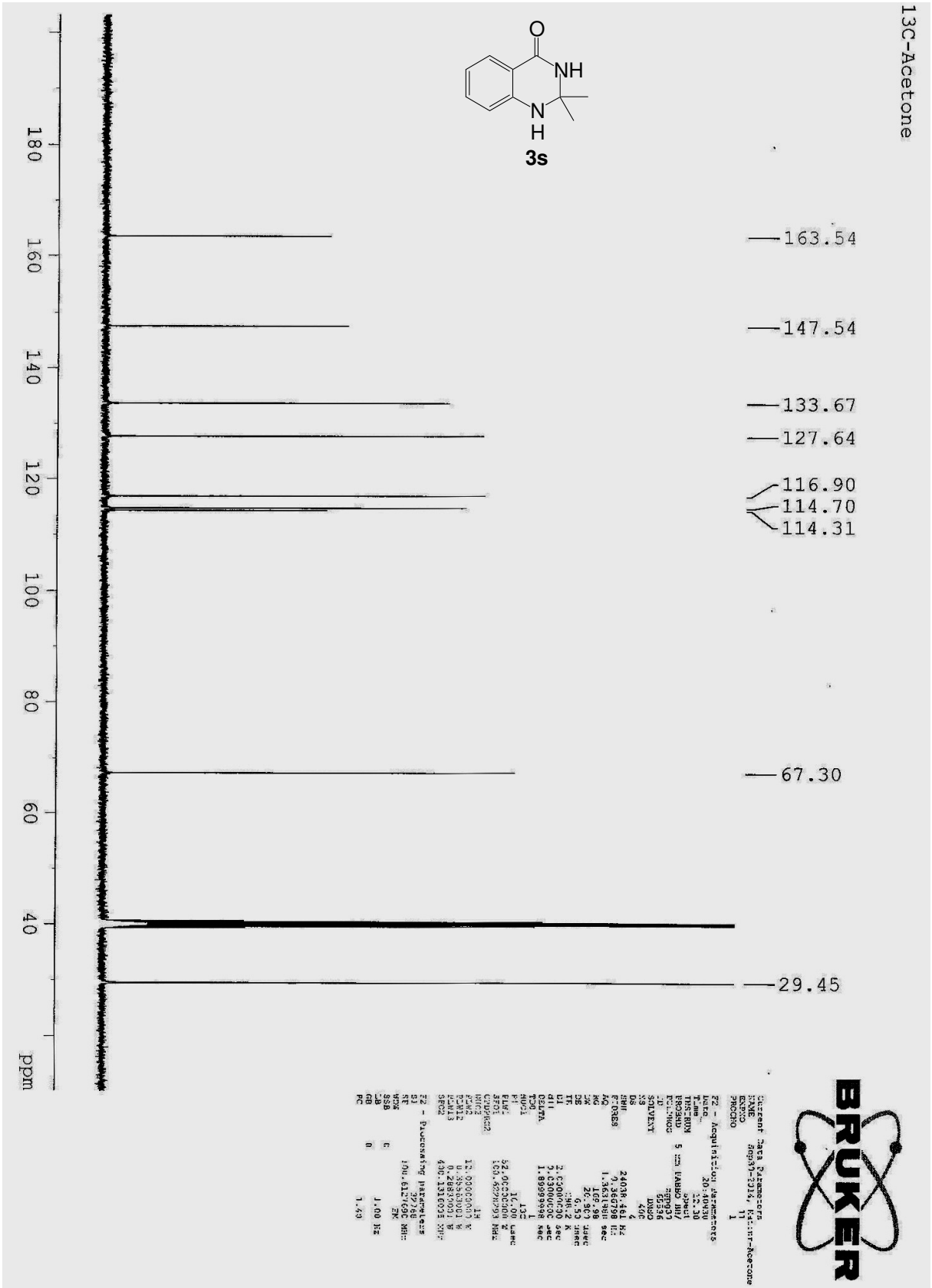

13C -aliphatic
O
NH
N
H
3r
165.57
147.50
133.83
128.54
119.32
115.96
114.74
65.14
37.65
17.40
13.82
180
160
140
120
100
80
60
40
20
0
ppm
Current Data Parameters
NAME
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141010
Time 11.17 h
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 400
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631598 sec
RG 20.800
DW 6.50 usec
DE 298.1 K
TE 2.0000000 sec
D1 0.0000000 sec
DELTA 1.8999998 sec
TD0 1
NUC1 13C
P1 10.00 usec
PL1 52.0000000 W
SFO1 100.6228293 MHz
CPDPRG2
NUC2 1H
PLW2 12.0900000 W
PLW12 0.5583000 W
PLW13 0.2883000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 65536
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1H-Acetone



BRUKER

```
Current Data Parameters
NAME      Sep30-2014, Matiur-Aceto
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140930
Time              12.05
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                65536
SOLVENT             DMSO
NS                    12
DS                     2
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894966 sec
RG                80.05
DW              62.400 usec
DE               6.50 usec
TE              298.1 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                1H
P1                15.50 usec
PLW1        12.00000000 W

F2 - Processing parameters
SI                65536
SF           400.1300000 MHz
WDW                 EM
SSB       0
LB              0.30 Hz
GB        0
PC                1.00
```


13C-Acetone
3s
163.54
147.54
133.67
127.64
116.90
114.70
114.31
67.30
29.45
ppm
180
160
140
120
100
80
60
40
BRUKER