Supporting Information

Iron-Catalyzed Annulations of 2-(2-Alkynyl)phenoxy)-1-arylethanones Leading to Substituted Naphthalen-1-ols

Zhi-Qiang Wang,[a] Yun Liang,[a] Yong Lei,[a] Ming-Bo Zhou,[a] and Jin-Heng Li*[a,b]

[a] *Key Laboratory of Chemical Biology & Traditional Chinese Medicine Research (Ministry of Education), Hunan Normal University, Changsha 410081, China*

[b] *College of Chemistry and Materials Science, Wenzhou University, Wenzhou 325035, China*

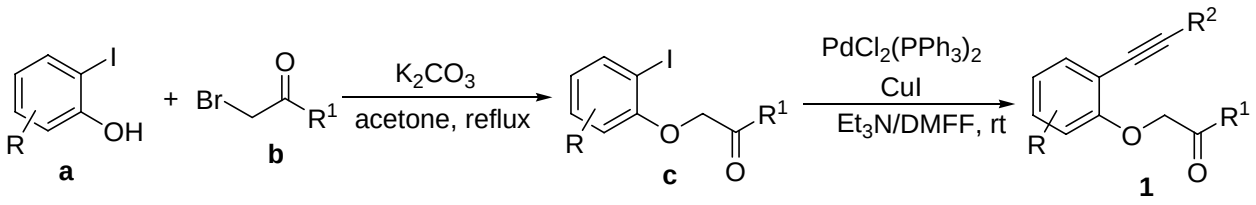
jhli@hunnu.edu.cn

List of Contents

(A) Typical Experimental Procedure

(a) Materials:

Substrates **1** were prepared according to the known procedures.[1]



(c) Typical Experimental Procedures:

(i) Typical Experimental Procedure for the Fe-Catalyzed Annulation of 2-(2-Alkynyl)phenoxy)-1-arylethanones (1):

A mixture of 2-(2-ethynyl)phenoxy)-1-arylethanone **1** (0.2 mmol), $FeCl_3$ (20 mol %), KOAc (4 equiv), 4 Å Molecular Sieve (100 mg) and anhydrous MeCN (2 mL) was stirred in a Schlenk tube at 120 ^oC (oil bath temperature) under argon atmosphere until complete consumption of starting material as monitored by TLC and GC-MS analysis. Then the mixture was filtered by a crude column, washed with diethyl ether, and evaporated under vacuum. The residue was purified by flash column chromatography (hexane/ethyl acetate) to afford the pure product **2**.

(B) Analytical data for 1 and 2

1-Phenyl-2-(2-(2-phenylethynyl)phenoxy)ethanone (1a):

Brown solid, mp 60.5-62.1 ^oC (uncorrected); ^1H NMR (500 MHz, $CDCl_3$) δ: 8.06 (d, J = 7.5 Hz, 2H), 7.57 (t, J = 7.5 Hz, 1H), 7.51 (d, J = 7.5 Hz, 1H), 7.44-7.47 (m, 4H), 7.30-7.31 (m, 3H), 7.24-7.27 (m, 1H), 6.98 (t, J = 7.5 Hz, 1H), 6.85 (d, J = 8.0 Hz, 1H), 5.34 (s, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 194.6, 158.5, 134.7, 133.8, 133.6, 131.6, 129.7, 128.8, 128.5, 128.3, 128.2, 123.5, 121.7, 113.5, 11.9, 94.0, 85.5, 72.1; HRMS (EI) for $C_{22}H_{16}O_2$ (M^+): calcd. 312.1150, found 312.1148.

1-Phenyl-2-(2-(2-*m*-tolylethynyl)phenoxy)ethanone (1b)

Brown solid, mp 79.6-82.1 ^{o}C (uncorrected); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.11 (d, *J* = 7.5 Hz, 2H), 7.61-7.62 (m, 1H), 7.48-7.55 (m, 3H), 7.27-7.31 (m, 3H), 7.23 (t, *J* = 8.0 Hz, 1H), 7.15 (d, *J* = 7.5 Hz, 1H), 7.01 (t, *J* = 7.5 Hz, 1H), 6.89 (d, *J* = 8.5 Hz, 1H), 5.37 (s, 2H), 2.35 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 194.7, 158.5, 137.9, 134.7, 133.8, 133.6, 132.2, 129.6, 129.1, 128.8, 128.7, 128.6, 128.2, 123.2, 121.7, 113.6, 113.0, 94.2, 85.2, 72.1, 21.3; HRMS (EI) for $C_{23}H_{18}O_2$ (M^+): calcd. 326.1307, found 326.1307.

1-Phenyl-2-(2-(2-*o*-tolylethynyl)phenoxy)ethanone (1c)

Brown solid, mp 66.5-68.4 ^{o}C (uncorrected); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.05 (d, *J* = 8.0 Hz, 2H), 7.55-7.58 (m, 1H), 7.51 (d, *J* = 7.5 Hz, 2H), 7.43-7.46 (m, 3H), 7.20-7.27 (m, 3H), 7.12-7.14 (m, 1H), 6.98 (t, *J* = 7.5 Hz, 1H), 6.86 (d, *J* = 8.5 Hz, 1H), 5.33 (s, 2H), 2.46 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 194.6, 158.4, 140.3, 134.6, 133.9, 133.6, 131.9, 129.6, 129.4, 128.8, 128.5, 128.2, 125.5, 123.3, 121.7, 113.7, 112.8, 93.0, 89.4, 72.0, 20.7; HRMS (EI) for $C_{23}H_{18}O_2$ (M^+): calcd. 326.1307, found 326.1307.

2-(2-(2-(4-Fluorophenyl)ethynyl)phenoxy)-1-phenylethanone (1d)

Brown solid, mp 118.8-120.6 ^{o}C (uncorrected); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.06 (d, *J* = 7.0 Hz, 2H), 7.57-7.60 (m, 1H), 7.42-7.51 (m, 5H), 7.26-7.28 (m, 1H), 6.97-7.02 (m, 3H), 6.85 (d, *J* = 7.0 Hz, 1H), 5.34 (s, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 194.4, 161.5 (d, *J* = 234.0 Hz, 1C), 158.4, 134.5, 133.8, 133.5, 133.4, 129.6, 128.7, 128.4, 121.6, 119.5, 115.5 (d, *J* = 25.0 Hz, 1C), 113.2, 112.7, 92.8, 85.1, 71.8; HRMS (EI) for $C_{22}H_{15}FO_2$ (M^+): calcd. 330.1056, found 330.1056.

1-Phenyl-2-(2-(2-(4-(trifluoromethyl)phenyl)ethynyl)phenoxy)ethanone (1e):

Brown solid, mp 77.1-78.3 ^{o}C (uncorrected); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.05 (d, *J* = 7.5 Hz, 2H), 7.46-7.61 (m, 8H), 7.30 (t, *J* = 7.5 Hz, 1H), 7.00 (t, *J* = 7.5 Hz, 1H), 6.85 (d, *J* = 8.5 Hz, 1H), 5.35 (s, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 194.2, 158.6, 134.5, 133.9, 133.6, 131.7, 130.2, 129.8, 129.5, 128.8,

128.4, 127.3 (q, J = 273.8.0 Hz, 1C), 125.1 (d, J = 3.6 Hz, 1C), 122.9, 121.6, 112.6 (d, J = 8.8 Hz, 1C), 92.5, 87.9, 71.7; HRMS (EI) for $C_{23}H_{15}F_3O_2$ (M^+): calcd. 380.1024, found 380.1024.

1-Phenyl-2-(2-(2-(thiophen-2-yl)ethynyl)phenoxy)ethanone (1f)

Brown oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.05 (d, J = 8.0 Hz, 2H), 7.58 (t, J = 7.5 Hz, 1H), 7.45-7.49 (m, 3H), 7.21-7.27 (m, 3H), 6.95-7.06 (m, 2H), 6.83 (d, J = 8.5 Hz, 1H), 5.34 (s, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 194.6, 158.4, 134.5, 133.9, 133.5, 131.9, 129.8, 128.8, 128.5, 127.3, 127.1, 123.5, 121.7, 113.1, 112.9, 89.3, 87.1, 72.0; HRMS (EI) for $C_{20}H_{14}O_2S$ (M^+): calcd. 318.0715, found 318.0714.

2-(2-(Oct-1-ynyl)phenoxy)-1-phenylethanone (1g)

Brown solid, mp 57.1-59.4 ^{o}C (uncorrected); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.04 (d, J = 8.0 Hz, 2H), 7.59-7.62 (m, 1H), 7.48 (t, J = 7.5 Hz, 2H), 7.38 (t, J = 7.5 Hz, 1H), 7.18 (t, J = 8.0 Hz, 1H), 6.92 (t, J = 7.0 Hz, 1H), 5.32 (s, 2H), 2.41 (t, J = 7.0 Hz, 2H), 1.54-1.58 (m, 2H), 1.41-1.44 (m, 2H), 1.26-1.30 (m, 4H), 0.89 (t, J = 7.2 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 194.7, 158.5, 134.6, 133.8, 128.7, 128.4, 121.7, 114.3, 113.2, 95.3, 76.4, 72.1, 31.4, 28.8, 28.7, 22.6, 19.8, 14.1; HRMS (EI) for $C_{22}H_{24}O_2$ (M^+): calcd. 320.1776, found 320.1773.

2-(2-(3,3-Dimethylbut-1-ynyl)phenoxy)-1-phenylethanone (1h)

Yellow solid, mp 60.8-62.7 ^{o}C (uncorrected); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.04 (d, J = 8.0 Hz, 2H), 7.60 (t, J = 7.5 Hz, 1H), 7.47-7.50 (m, 2H), 7.36 (d, J = 7.5 Hz, 1H), 7.19 (t, J = 8.0 Hz, 1H), 6.92 (t, J = 7.5 Hz, 1H), 6.85 (d, J = 8.5 Hz, 1H), 5.28 (s, 2H), 1.27 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 194.8, 158.4, 134.7, 133.7, 133.6, 128.7, 128.7, 128.5, 121.7, 114.3, 113.6, 103.3, 74.8, 72.3, 30.9, 28.1; HRMS (EI) for $C_{20}H_{20}O_2$ (M^+): calcd. 292.1463, found 292.1463.

1-Phenyl-2-(2-(2-(trimethylsilyl)ethynyl)phenoxy)ethanone (1i)

White solid, mp 75.3-76.9 °C (uncorrected); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.05 (d, *J* = 8.0 Hz, 2H), 7.59-7.62 (m, 1H), 7.45-7.49 (m, 2H), 7.43 (t, *J* = 1.5 Hz, 1H), 7.22-7.25 (m, 1H), 6.91-6.95 (m, 1H), 6.83 (d, *J* = 8.0 Hz, 1H), 5.32 (s, 2H), 0.21 (t, *J* = 4.0 Hz, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 194.6, 158.9, 134.6, 134.2, 133.8, 129.8, 128.7, 128.5, 121.6, 113.3, 100.8, 99.1, 72.1, -0.1; HRMS (EI) for $C_{19}H_{20}O_2Si$ (M^+): calcd. 308.1233, found 308.1232.

2-(5-Methyl-2-(2-phenylethynyl)phenoxy)-1-phenylethanone (1j)

Brown solid, mp 87.8-90.1 °C (uncorrected); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.07-8.08 (m, 2H), 7.57 (d, *J* = 7.5 Hz, 1H), 7.44-7.47 (m, 5H), 7.38-7.40 (m, 3H), 5.34 (s, 2H), 2.33 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 194.7, 158.4, 140.2, 134.6, 133.7, 133.3, 131.5, 128.7, 128.5, 128.2, 127.9, 123.6, 122.5, 113.8, 110.4, 93.3, 85.7, 72.0, 21.8; HRMS (EI) for $C_{23}H_{18}O_2$ (M^+): calcd. 326.1307, found 326.1307.

2-(2-(2-Phenylethynyl)phenoxy)-1-*p*-tolylethanone (1k)

Brown solid, mp 91.5-93.0 °C (uncorrected); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.96 (d, *J* = 8.0 Hz, 2H), 7.50 (d, *J* = 7.5 Hz, 1H), 7.44-7.45 (m, 2H), 7.23-7.30 (m, 6H), 6.97 (t, *J* = 7.5 Hz, 1H), 6.84 (d, *J* = 8.5 Hz, 1H), 5.30 (s, 2H), 2.37 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 194.2, 158.5, 144.7, 133.5, 132.1, 131.6, 129.6, 129.4, 128.6, 128.2, 128.1, 123.4, 121.5, 113.3, 112.7, 93.9, 85.5, 71.9, 21.7; HRMS (EI) for $C_{23}H_{18}O_2$ (M^+): calcd. 326.1307, found 326.1307.

1-(2-Methoxyphenyl)-2-(2-(2-phenylethynyl)phenoxy)ethanone (1l)

Brown solid, mp 77.9-79.2 °C (uncorrected); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.92 (d, *J* = 8.0 Hz, 2H), 7.47-7.51 (m, 4H), 7.30-7.33 (m, 3H), 7.22-7.25 (m, 1H), 7.04 (t, *J* = 7.5 Hz, 2H), 6.93-6.97 (m, 2H), 6.77 (d, *J* = 8.0 Hz, 1H), 5.33 (s, 2H), 3.88 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 195.4, 159.3, 159.0, 134.6, 133.5, 131.6, 130.9, 129.5, 128.1, 128.0, 125.0, 123.5, 121.1, 121.0, 113.0, 112.9, 111.5, 93.6, 85.7, 75.1, 55.6; HRMS (EI) for $C_{23}H_{18}O_3$ (M^+): calcd. 342.1256, found 342.1256.

1-(3-Bromophenyl)-2-(2-(2-phenylethynyl)phenoxy)ethanone (1m)

Yellow oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.17-8.19 (m, 1H), 8.00 (d, *J* = 8.0 Hz, 1H), 7.67 (d, *J* = 8.0 Hz, 1H), 7.51 (d, *J* = 8.0 Hz, 1H), 7.44-7.46 (m, 2H), 7.25-7.32 (m, 5H), 6.99 (t, *J* = 7.5 Hz, 1H), 6.85 (d, *J* = 8.0 Hz, 1H), 5.28 (s, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 193.6, 158.2, 136.6, 136.2, 133.6, 131.6, 131.5, 130.3, 129.6, 128.2, 128.2, 127.2, 123.3, 123.0, 121.9, 113.4, 112.8, 94.1, 85.2, 72.0; HRMS (EI) for $C_{22}H_{15}BrO_2$ (M^+): calcd. 390.0255, found 390.0253.

1-(Naphthalen-2-yl)-2-(2-(2-phenylethynyl)phenoxy)ethanone (1n)

Yellow solid, mp 101.7-103.3 ^{o}C (uncorrected); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.67 (br, 1H), 7.85-8.07 (br, 4H), 7.25-7.58 (br, 8H), 6.92-6.98 (br, 2H), 5.45 (s, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 194.7, 158.4, 135.8, 133.6, 132.4, 131.9, 131.6, 131.5, 130.7, 129.8, 129.6, 128.8, 128.7, 128.6, 128.5, 128.1, 128.1, 127.7, 126.8, 123.9, 123.3, 121.7, 113.4, 112.8, 94.0, 85.5, 72.2; HRMS (EI) for $C_{26}H_{18}O_2$ (M^+): calcd. 362.1307, found 362.1307.

1-Phenyl-2-(2-(2-phenylethynyl)phenoxy)propan-1-one (1o)

Yellow solid, mp 94.9-96.7 ^{o}C (uncorrected); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.16 (d, *J* = 8.5 Hz, 1H), 7.47-7.53 (m, 4H), 7.39-7.42 (m, 2H), 7.33-7.36 (m, 3H), 7.17-7.20 (m, 1H), 6.91-6.94 (m, 1H), 6.82 (d, *J* = 8.0 Hz, 1H), 5.49-5.53 (m, 1H), 1.79 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 198.9, 158.0, 133.9, 133.6, 133.5, 131.5, 129.6, 129.2, 128.6, 128.3, 128.1, 123.5, 121.6, 114.1, 113.8, 93.8, 85.8, 78.8, 18.9; HRMS (EI) for $C_{23}H_{18}O_2$ (M^+): calcd. 326.1307, found 326.1306.

3-(2-Hydroxyphenyl)-4-phenylnaphthalen-1-ol (2a)

Yellow solid, mp 213.2-214.9 ^{o}C (uncorrected); ^{1}H NMR (500 MHz, DMSO-D_6) δ: 10.22 (s, 1H), 9.18 (s, 1H), 8.21 (d, *J* = 8.5 Hz, 1H), 7.43-7.46 (m, 1H), 7.38-7.39 (m, 2H), 7.22-7.25 (m, 2H), 7.15-7.19 (m, 3H), 6.95 (t, *J* = 8.0 Hz, 1H), 6.82 (s, 1H), 6.78 (d, *J* = 9.0 Hz, 1H), 6.73 (d, *J* = 9.0 Hz, 1H), 6.54 (t, *J* = 7.5 Hz, 1H); ^{13}C NMR (125 MHz, DMSO-D_6) δ: 154.3, 151.6, 139.2, 136.2, 133.3, 131.2, 130.9, 129.0,

128.9, 127.8, 127.4, 126.2, 126.2, 125.6, 124.2, 123.7, 121.9, 118.1, 115.1, 110.8; IR (KBr, cm^{-1}): 3502, 3355, 1596; LRMS (EI, 70 eV) *m/z* (%): 312 (M^+, 100), 295 (6), 265 (9), 178 (6), 132 (11), 44 (22), 32 (55); HRMS (EI) for $C_{22}H_{16}O_2$ (M^+): calcd. 312.1150, found 312.1150.

3-(2-Hydroxyphenyl)-4-*m*-tolylnaphthalen-1-ol (2b)

Yellow solid, mp 89.7-91.3 °C (uncorrected); ^{1}H NMR (500 MHz, DMSO-D_6) δ: 10.15 (s, 1H), 9.14 (s, 1H), 8.21 (d, *J* = 8.5 Hz, 1H), 7.36-7.45 (m, 3H), 7.11 (t, *J* = 7.5 Hz, 1H), 6.93-6.70 (m, 4H), 6.74-6.81 (m, 3H), 6.55 (t, *J* = 7.0 Hz, 1H), 2.21 (s, 3H); ^{13}C NMR (125 MHz, DMSO-D_6) δ: 154.9, 152.0, 139.6, 136.6, 136.6, 133.8, 132.1, 131.7, 129.5, 129.5, 128.2, 127.8, 127.4, 126.6, 126.2, 124.6, 124.3, 122.4, 118.6, 115.5, 111.3, 21.5; IR (KBr, cm^{-1}): 3206, 1594; LRMS (EI, 70 eV) *m/z* (%): 326 (M^+, 100), 311 (6), 281 (4), 252 (4), 239 (4), 205 (4), 178 (3), 132 (9), 40 (83); HRMS (EI) for $C_{23}H_{18}O_2$ (M^+): calcd. 326.1307, found 326.1306.

3-(2-Hydroxyphenyl)-4-*o*-tolylnaphthalen-1-ol (2c)

Yellow solid, mp 181.5-183.7 °C (uncorrected); ^{1}H NMR (500 MHz, DMSO-D_6) δ: 10.15 (s, 1H), 9.19 (s, 1H), 8.22 (d, *J* = 8.0 Hz, 1H), 7.44 (t, *J* = 7.5 Hz, 1H), 7.35 (t, *J* = 9.0 Hz, 1H), 7.04-7.14 (m, 5H), 6.93-6.96 (m, 1H), 6.87 (s, 1H), 6.75 (t, *J* = 9.0 Hz, 1H), 6.81 (d, *J* = 7.5 Hz, 1H), 6.53 (t, *J* = 7.5 Hz, 1H), 1.88 (s, 3H); ^{13}C NMR (125 MHz, DMSO-D_6) δ: 154.8, 152.0, 139.2, 137.5, 136.6, 133.5, 129.7, 129.2, 128.5, 128.3, 127.2, 126.7, 125.9, 125.4, 124.6, 124.3, 122.5, 118.5, 115.6, 111.6, 20.2; IR (KBr, cm^{-1}): 3506, 3368, 1595; LRMS (EI, 70 eV) *m/z* (%): 326 (M^+, 100), 307 (7), 265 (5), 215 (6), 178 (3), 132 (8), 40 (14); HRMS (EI) for $C_{23}H_{18}O_2$ (M^+): calcd. 326.1307, found 326.1307.

4-(4-Fluorophenyl)-3-(2-hydroxyphenyl)naphthalen-1-ol (2d)

Yellow solid, mp 146.1-149.7 °C (uncorrected); ^{1}H NMR (500 MHz, DMSO-D_6) δ: 10.24 (s, 1H), 9.17 (s, 1H), 8.24 (d, *J* = 8.0 Hz, 1H), 7.44-7.47 (m, 1H), 7.39 (d, *J* = 4.0 Hz, 2H), 7.14-7.17 (m, 2H), 7.06 (t, *J* = 9.0 Hz, 2H), 6.98 (t, *J* = 8.0 Hz, 1H), 6.84 (s, 1H), 6.81 (d, *J* = 7.5 Hz, 1H), 6.75 (d, *J* = 8.0 Hz, 1H),

6.59 (t, *J* = 7.5 Hz, 1H); ^{13}C NMR (125 MHz, DMSO-D_6) δ: 161.3 (d, *J* = 233.8 Hz, 1C), 154.8, 152.3, 137.0, 135.9 (d, *J* = 3.8 Hz, 1C), 133.8, 133.2, 131.7, 129.3, 128.4 (d, *J* = 3.8 Hz, 1C), 126.8, 125.9, 124.7, 124.3, 122.5, 118.7, 115.7, 114.8, 114.7, 111.2; IR (KBr, cm^{-1}): 3422, 3064, 1591, 1510; LRMS (EI, 70 eV) m/z (%): 330 (M^+, 100), 313 (5), 283 (7), 235 (3), 207 (5), 183 (3), 141 (6), 40 (45); HRMS (EI) for $C_{22}H_{15}FO_2$ (M^+): calcd. 330.1056, found 330.1053.

4-(4-(Trifluoromethyl)phenyl)-3-(2-hydroxyphenyl)naphthalen-1-ol (2e)

Yellow solid, mp 143.7-144.4 °C (uncorrected); ^{1}H NMR (500 MHz, DMSO-D_6) δ: 10.33 (s, 1H), 9.21 (s, 1H), 8.24 (d, *J* = 8.0 Hz, 1H), 7.60 (d, *J* = 9.0 Hz, 2H), 7.33-7.49 (m, 5H), 6.99 (t, *J* = 9.0 Hz, 1H), 6.84 (s, 1H), 6.83 (t, *J* = 7.5 Hz, 1H), 6.73 (d, *J* = 9.0 Hz, 1H), 6.59 (t, *J* = 9.0 Hz, 1H); ^{13}C NMR (125 MHz, DMSO-D_6) δ: 167.5, 154.8, 152.7, 144.3, 136.9, 133.3, 132.3, 132.0, 131.7, 128.9, 128.6, 127.9, 127.4, 127.1, 127.1, 126.0, 125.6, 124.9, 124.7, 124.3, 122.6, 118.7, 115.7, 111.2; IR (KBr, cm^{-1}): 3432, 2926, 1727, 1592; LRMS (EI, 70 eV) *m/z* (%): 380 (M^+, 66), 361 (5), 292 (2), 149 (6), 132 (5), 40 (100); HRMS (EI) for $C_{23}H_{15}F_3O_2$ (M^+): calcd. 380.1024, found 380.1023.

3-(2-Hydroxyphenyl)-4-(thiophen-2-yl)naphthalen-1-ol (2f)

Yellow solid, mp 196.5-198.2 °C (uncorrected); ^{1}H NMR (500 MHz, DMSO-D_6) δ: 10.35 (s, 1H), 9.21 (s, 1H), 8.20 (d, *J* = 8.5 Hz, 1H), 7.59 (d, *J* = 7.5 Hz, 1H), 7.41-7.48 (m, 3H), 6.96-7.02 (m, 2H), 6.87-6.89 (m, 2H), 6.82 (s, 1H), 6.77 (d, *J* = 9.0 Hz, 1H), 6.60 (t, *J* = 7.5 Hz, 1H); ^{13}C NMR (125 MHz, DMSO-D_6) δ: 154.9, 153.0, 140.1, 139.0, 134.8, 131.3, 129.1, 129.0, 128.5, 127.1, 126.9, 126.6, 126.0, 124.9, 124.2, 122.4, 121.2, 118.6, 115.6, 111.3; IR (KBr, cm^{-1}): 3461, 3356, 2917, 2849; LRMS (EI, 70 eV) *m/z* (%): 318 (M^+, 100), 301 (8), 285 (26), 271 (33), 255 (9), 189 (5), 136 (9), 113 (10), 40 (83); HRMS (EI) for $C_{20}H_{14}O_2S$ (M^+): calcd. 318.0715, found 318.0711.

4-Hexyl-3-(2-hydroxyphenyl)naphthalen-1-ol (2g)

Yellow oil; ^{1}H NMR (500 MHz, DMSO-D_6) δ: 9.85 (s, 1H), 9.21 (s, 1H), 8.16 (d, J = 8.0 Hz, 1H), 7.96 (d, J = 8.0 Hz, 1H), 7.52 (t, J = 8.0 Hz, 1H), 7.43 (t, J = 8.5 Hz, 1H), 7.19 (t, J = 7.5 Hz, 1H), 7.04 (d, J = 7.0 Hz, 1H), 6.93 (d, J = 9.0 Hz, 1H), 6.85 (t, J = 8.0 Hz, 1H), 6.61 (s, 1H), 2.78-2.81 (m, 1H), 2.64-2.65 (m, 1H), 1.48~1.55 (m, 1H), 1.32-1.37 (m, 1H), 1.04-1.23 (m, 6H), 0.76 (t, J = 7.0 Hz, 3H); ^{13}C NMR (125 MHz, DMSO-D_6) δ: 154.7, 150.9, 136.5, 133.0, 131.2, 129.7, 128.6, 127.3, 126.5, 124.8, 124.5, 124.2, 122.8, 119.1, 115.9, 111.3, 31.2, 30.8, 29.3, 28.9, 22.4, 14.4; IR (KBr, cm^{-1}): 3176, 3067, 2954, 2927, 1594; LRMS (EI, 70 eV) *m/z* (%): 320 (M^+, 26), 249 (71), 231 (13), 202 (7), 40 (100); HRMS (EI) for $C_{22}H_{24}O_2$ (M^+): calcd. 320.1776, found 320.1776.

4-*tert*-Butyl-3-(2-hydroxyphenyl)naphthalen-1(4*H*)-one (2h)

Yellow oil; ^{1}H NMR (500 MHz, DMSO-D_6) δ: 9.93 (s, 1H), 7.91 (d, J = 7.5 Hz, 1H), 7.56 (d, J = 4.0 Hz, 2H), 7.44-7.47 (m, 1H), 7.38 (d, J = 7.5 Hz, 1H), 7.21 (t, J = 8.5 Hz, 1H), 6.93 (d, J = 8.0 Hz, 1H), 6.88 (t, J = 7.5 Hz, 1H), 6.59 (s, 1H), 4.46 (s, 1H), 0.68 (s, 9H); ^{13}C NMR (125 MHz, DMSO-D_6) δ: 186.4, 154.5, 144.1, 133.9, 130.7, 130.5, 130.5, 130.2, 129.6, 129.4, 127.3, 125.2, 119.8, 116.8, 53.7, 38.3, 28.9; IR (KBr, cm^{-1}): 3178, 3068, 2958, 2927, 1636; LRMS (EI, 70 eV) *m/z* (%): 292 (M^+, 1), 250 (1), 236 (31), 40 (100); HRMS (EI) for $C_{20}H_{20}O_2$ (M^+): calcd. 292.1463, found 292.1461.

3-(2-Hydroxyphenyl)naphthalen-1-ol (2i)

Brown oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.21-8.23 (m, 1H), 7.83-7.84 (m, 1H), 7.51-7.57 (m, 1H), 7.27-7.33 (m, 2H), 7.00-7.04 (m, 2H), 6.91 (d, J = 1.5 Hz, 1H), 5.50-5.70 (br, 1H), 5.76-5.84 (br, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 152.5, 152.5, 134.9, 134.7, 130.2, 129.3, 127.8, 127.8, 127.3, 125.8, 123.8, 121.7, 120.8, 120.3, 115.8, 109.7; IR (KBr, cm^{-1}): 3524, 3313, 2917, 2850; LRMS (EI, 70 eV) m/z (%): 236 (M^+, 100), 219 (9), 178 (11), 152 (5), 118 (6), 73 (9); HRMS (EI) for $C_{16}H_{12}O_2$ (M^+): calcd. 236.0837, found 236.0835.

3-(2-Hydroxy-4-methylphenyl)-4-phenylnaphthalen-1-ol (2j)

Yellow solid, mp 77.2-80.1 ^{o}C (uncorrected); ^{1}H NMR (500 MHz, DMSO-D_6) δ: 10.14 (s, 1H), 9.01 (s, 1H), 8.20 (d, J = 8.0 Hz, 1H), 7.43 (t, J = 8.5 Hz, 1H), 7.35-7.37 (m, 2H), 7.13-7.25 (m, 5H), 6.80 (s, 1H), 6.65 (d, J = 8.0 Hz, 1H), 6.54 (s, 1H), 6.35 (d, J = 7.5 Hz, 1H), 2.13 (s, 3H); ^{13}C NMR (125 MHz, DMSO-D_6) δ:154.6, 152.0, 139.8, 137.3, 136.7, 133.8, 131.6, 131.5, 129.5, 128.0, 126.7, 126.6, 126.6, 126.1, 124.5, 124.2, 122.4, 119.4, 116.2, 111.5, 21.3; IR (KBr, cm^{-1}): 3191, 2921, 2850, 1593; LRMS (EI, 70 eV) *m/z* (%): 326 (M^+, 10), 316 (2), 249 (1), 40 (100); HRMS (EI) for $C_{23}H_{18}O_2$ (M^+): calcd. 326.1307, found 326.1306.

3-(2-Hydroxyphenyl)-6-methyl-4-phenylnaphthalen-1-ol (2k)

Yellow solid, mp 190.1-192.6 ^{o}C (uncorrected); ^{1}H NMR (500 MHz, DMSO-D_6) δ: 10.10 (s, 1H), 9.12 (s, 1H), 8.13 (d, J = 8.5 Hz, 1H), 7.14-7.30 (m, 7H), 6.94 (t, J = 8.0 Hz, 1H), 6.73~6.78 (m, 3H), 6.53 (t, J = 7.5 Hz, 1H), 2.31 (s, 3H); ^{13}C NMR (125 MHz, DMSO-D_6) δ: 154.8, 152.1, 139.8, 136.8, 135.6, 134.0, 131.8, 131.5, 129.6, 128.9, 128.2, 127.9, 126.7, 126.6, 125.1, 122.6, 122.4, 118.5, 115.6, 110.6, 22.1; IR (KBr, cm^{-1}): 3190, 3060, 2253, 1596; LRMS (EI, 70 eV) *m/z* (%): 326 (M^+, 100), 311 (7), 283 (4), 263 (15), 132 (10), 40 (36); HRMS (EI) for $C_{23}H_{18}O_2$ (M^+): calcd. 326.1307, found 326.1307.

3-(2-Hydroxyphenyl)-8-methoxy-4-phenylnaphthalen-1-ol (2l)

Yellow solid, mp 89.2-91.4 ^{o}C (uncorrected); ^{1}H NMR (500 MHz, DMSO-D_6) δ: 9.54 (s, 1H), 9.16 (s, 1H), 7.15-7.28 (m, 4H), 7.11 (d, J = 7.0 Hz, 2H), 6.93~6.99 (m, 3H), 6.78 (d, J = 7.0 Hz, 1H), 6.71~6.73 (m, 2H), 6.52 (t, J = 7.5 Hz, 1H), 4.06 (s, 3H); ^{13}C NMR (125 MHz, DMSO-D_6) δ: 156.6, 154.7, 153.0, 139.9, 138.1, 135.7, 131.6, 131.3, 129.7, 128.9, 128.4, 128.0, 126.8, 126.6, 119.9, 118.5, 115.6, 114.4, 113.3, 104.9, 56.9; IR (KBr, cm^{-1}): 3353, 3063, 1624; LRMS (EI, 70 eV) *m/z* (%): 342 (M^+, 18), 324 (8), 309 (2), 126 (3), 113 (2), 40 (100); HRMS (EI) for $C_{23}H_{18}O_3$ (M^+): calcd. 342.1256, found 342.1255.

7-Bromo-3-(2-hydroxyphenyl)-4-phenylnaphthalen-1-ol (2m) and 5-bromo-3-(2-hydroxyphenyl)-4-phenylnaphthalen-1-ol (2m′)

2m/2m′ = 1.2 : 1; Yellow oil; ^{1}H NMR (500 MHz, DMSO-D_6) δ: 10.46 (s, 0.55H), 10.44 (s, 0.45H), 9.20 (s, 0.45H), 9.12 (s, 0.55H), 8.33~8.35 (m, 1H), 7.78 (d, J = 7.5 Hz, 0.5H), 7.51 (d, J = 7.5 Hz, 0.45H), 7.30-7.34 (m, 1H), 6.49-7.44 (m, 10H); ^{13}C NMR (125 MHz, DMSO-D_6) δ: 154.8, 154.5, 152.3, 151.2, 140.6, 140.2, 139.0, 137.5, 134.9, 132.9, 132.3, 131.6, 131.3, 131.3, 130.8, 129.6, 129.6, 129.0, 128.9, 128.7, 128.4, 128.2, 128.0, 127.1, 126.9, 126.9, 126.7, 125.4, 125.2, 124.4, 123.1, 119.7, 118.6, 118.3, 118.2, 115.6, 115.4, 112.6, 111.8; IR (KBr, cm^{-1}): 3161, 1591; LRMS (EI, 70 eV) *m/z* (%): 393 (23), 392 (M^++2, 91), 391 (25), 390 (M^+, 92), 361 (5), 311 (22), 293 (21), 252 (18), 217 (55), 132 (40), 40 (100); HRMS (EI) for $C_{22}H_{15}{}^{79}BrO_2$ (M^+): calcd. 390.0255, found 390.0251.

3-(2-Hydroxyphenyl)-4-phenylanthracen-1-ol (2n) and 3-(2-hydroxyphenyl)-4-phenylphenanthren-1-ol (2n′)

2n/2n′ = 6.1 : 1; Yellow solid, mp 125.2-127.7 °C (uncorrected); ^{1}H NMR (500 MHz, DMSO-D_6) δ: 10.27 (s, 0.86H), 10.18 (s, 0.14H), 9.15 (s, 0.14H), 9.13 (s, 0.86H), 8.25 (d, J = 9.0 Hz, 1H), 7.88 (d, J = 8.0 Hz, 1H), 7.78 (d, J = 9.0 Hz, 1H), 7.32 (d, J = 8.0 Hz, 1H) 7.18-7.22 (m, 3H), 7.02-7.04 (m, 1H), 6.92-6.96 (m, 3H), 6.69-6.72 (m, 2H), 6.52 (t, J = 7.5 Hz, 1H); ^{13}C NMR (125 MHz, DMSO-D_6) δ: 154.7, 152.6, 143.4, 139.4, 134.0, 131.5, 131.3, 130.7, 130.2, 130.1, 130.0, 129.9, 128.9, 128.7, 128.4, 128.2, 128.1, 126.8, 126.3, 126.2, 124.9, 122.7, 121.0, 118.3, 115.4, 113.4; IR (KBr, cm^{-1}): 2924, 1450; LRMS (EI, 70 eV) *m/z* (%): 362 (M^+, 3), 312 (2), 169 (1), 117 (1), 40 (100); HRMS (EI) for $C_{26}H_{18}O_2$ (M^+): calcd. 362.1307, found 362.1305.

3-(2-Hydroxyphenyl)-2-methyl-4-phenylnaphthalen-1-ol (2o)

Yellow solid, mp 84.3-86.6 °C (uncorrected); ^{1}H NMR (500 MHz, DMSO-D_6) δ: 9.15 (s, 1H), 9.04 (s, 1H), 8.29 (d, J = 8.5 Hz, 1H), 7.44 (t, J = 8.0 Hz, 1H), 7.23-7.30 (m, 3H), 7.13-7.17 (m, 3H), 7.05-7.07 (m, 1H), 6.94 (t, J = 7.5 Hz, 1H), 6.69-6.74 (m, 2H), 6.53 (t, J = 7.5 Hz, 1H), 2.04 (s, 3H); ^{13}C NMR (125 MHz, DMSO-D_6) δ: 155.0, 149.1, 140.4, 138.2, 131.9, 131.5, 131.5, 130.8, 130.3, 128.4, 128.2, 128.0, 127.5, 126.6, 126.2, 125.5, 125.0, 124.8, 122.3, 118.8, 118.7, 115.2, 14.3; IR (KBr, cm^{-1}): 2923, 2853,

2252; LRMS (EI, 70 eV) *m/z* (%): 326 (M^+, 100), 311 (16), 231 (6), 194 (8), 132 (7), 113 (7), 40 (62); HRMS (EI) for $C_{23}H_{18}O_2$ (M^+): calcd. 326.1307, found 326.1307.

The product (3o)

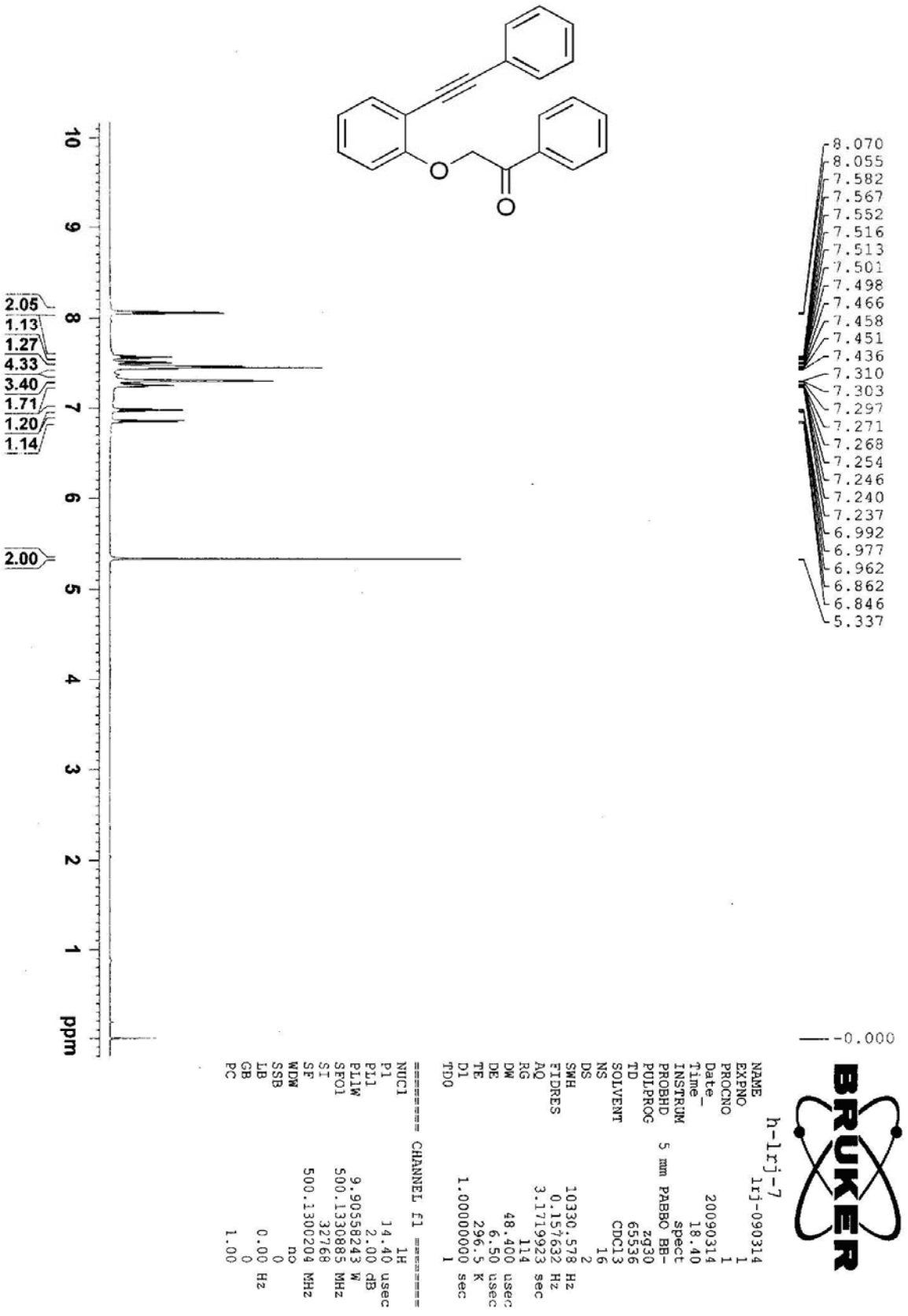
Yellow solid, mp 147.2-152.8 ^oC (uncorrected); ^1H NMR (500 MHz, $CDCl_3$) δ: 8.01 (d, *J* = 8.5 Hz, 1H), 7.60 (d, *J* = 5.0 Hz, 2H), 7.46-7.50 (m, 3H), 7.34 (t, *J* = 8.5 Hz, 1H), 7.19 (t, *J* = 8.5 Hz, 1H), 7.05−7.10 (m, 2H), 6.91 (d, *J* = 8.0 Hz, 1H), 6.69 (t, *J* = 7.5 Hz, 1H), 6.42 (d, *J* = 8.0 Hz, 1H), 1.75 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 200.1, 162.4, 142.9, 140.4, 136.4, 134.7, 131.1, 129.5, 128.7, 128.5, 128.3, 127.7, 127.5, 127.3, 126.9, 123.6, 123.4, 121.5, 111.8, 91.6, 26.5; IR (KBr, cm^{-1}): 1707; LRMS (EI, 70 eV) *m/z* (%): 324 (M^+, 92), 309 (27), 295 (15), 281 (25), 252 (20), 207 (19), 126 (7), 40 (100); HRMS (EI) for $C_{23}H_{16}O_2$ (M^+): calcd 324.1150, found 324.1150.

(C) References

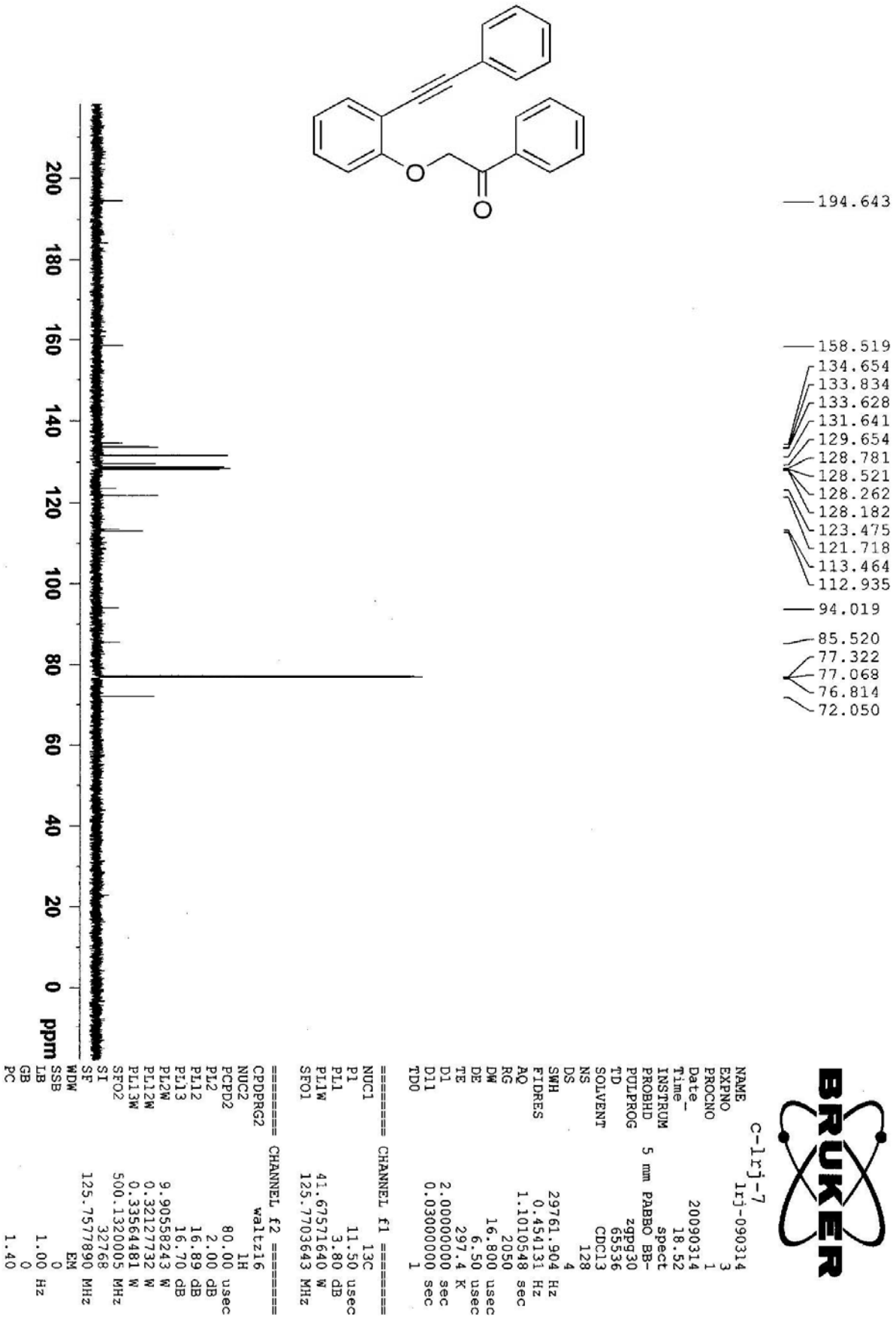
(1) (a) Bi, H.-P; Guo, L.-N.; Gou, F.-R.; Duan, X.-H.; Liu, X.-Y.; Liang, Y.-M. *J. Org. Chem.* **2007**, *73*, 4713. (b) Portscheller, J. L.; Malinakova, H. C. *Org. Lett.* **2002**, *4*, 3679.

(D) Spectra

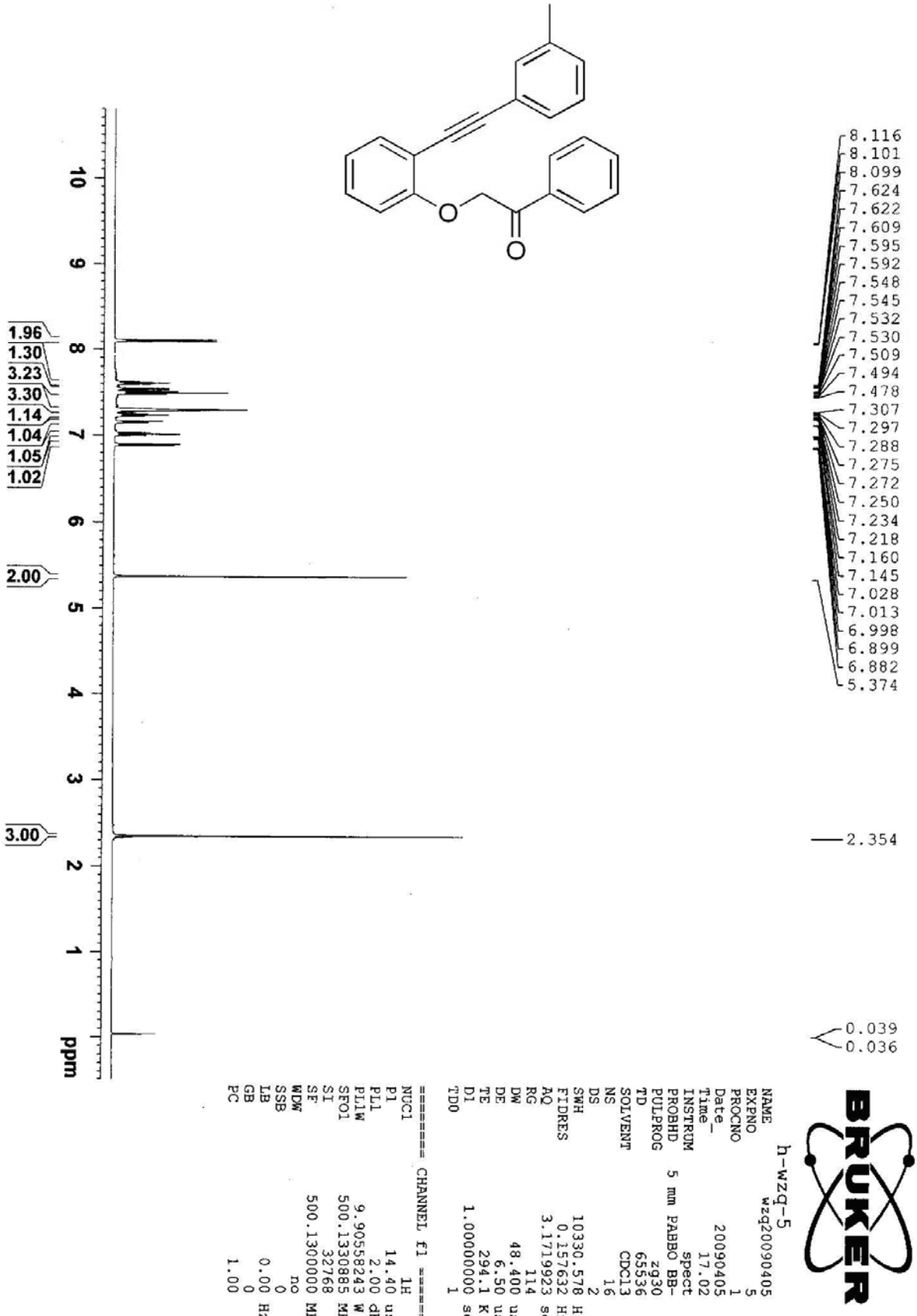
1-Phenyl-2-(2-(2-phenylethynyl)phenoxy)ethanone (1a)



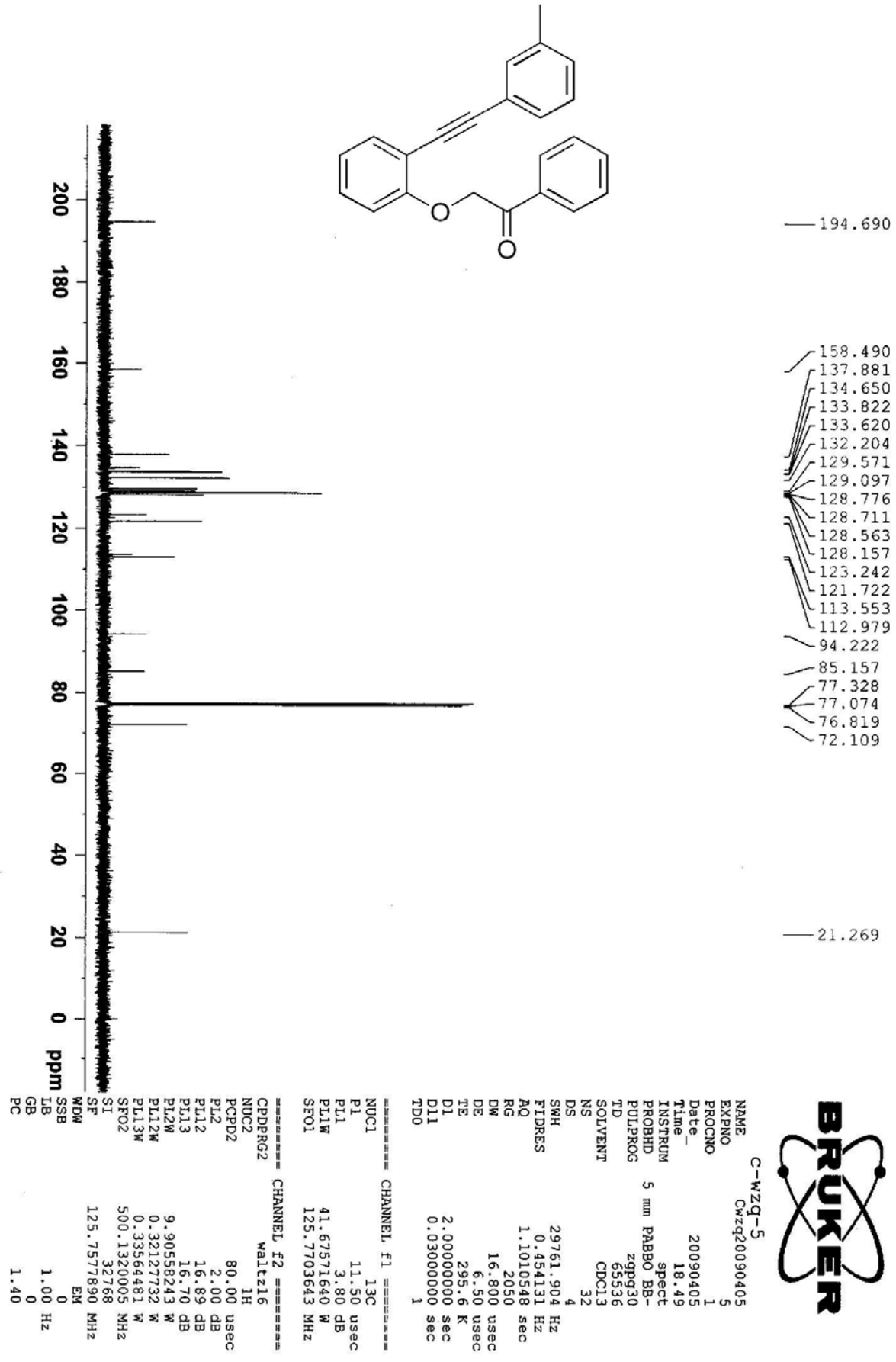
1-Phenyl-2-(2-(2-phenylethynyl)phenoxy)ethanone (1a)



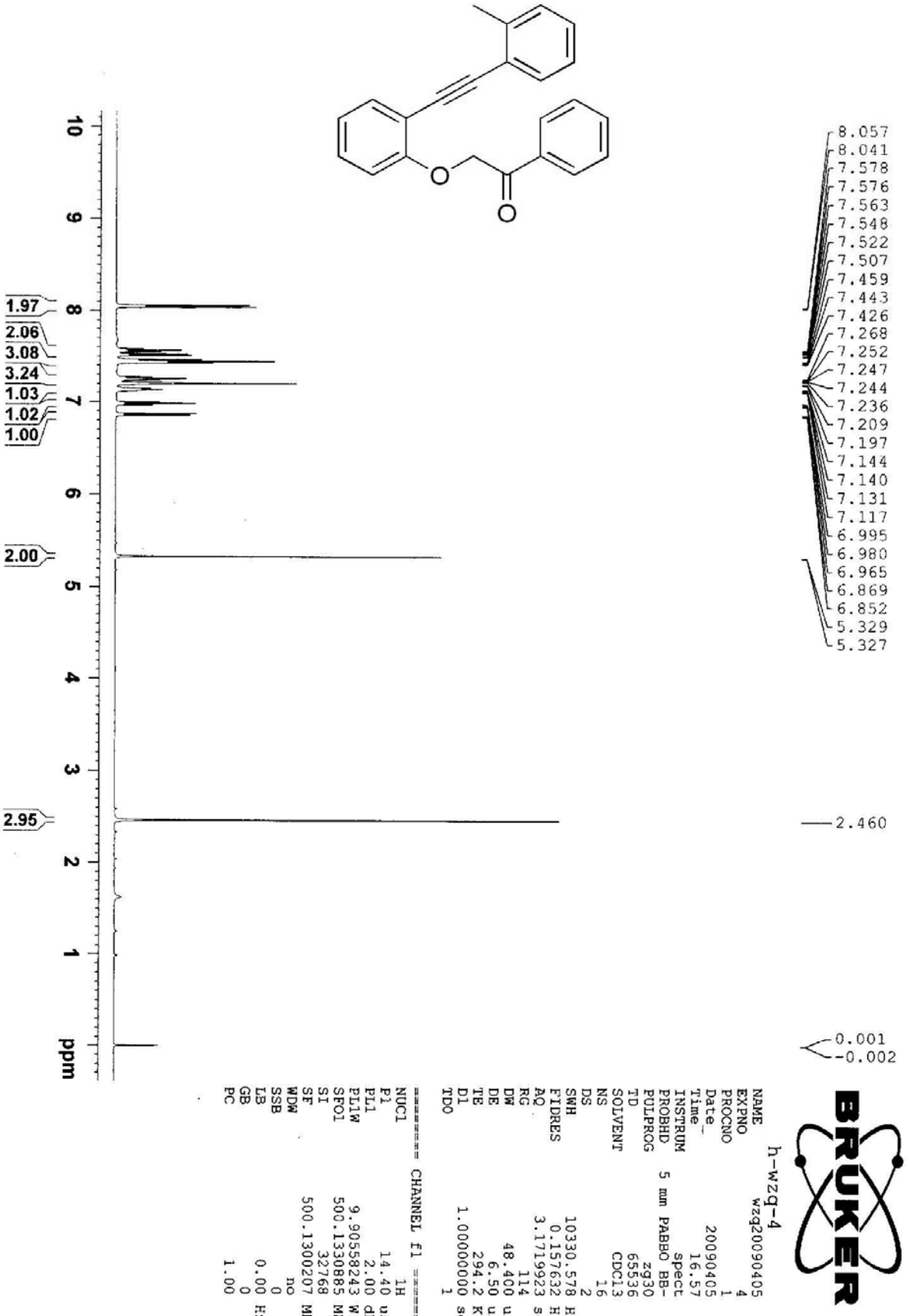
1-Phenyl-2-(2-(2-*m*-tolylethynyl)phenoxy)ethanone (1b)



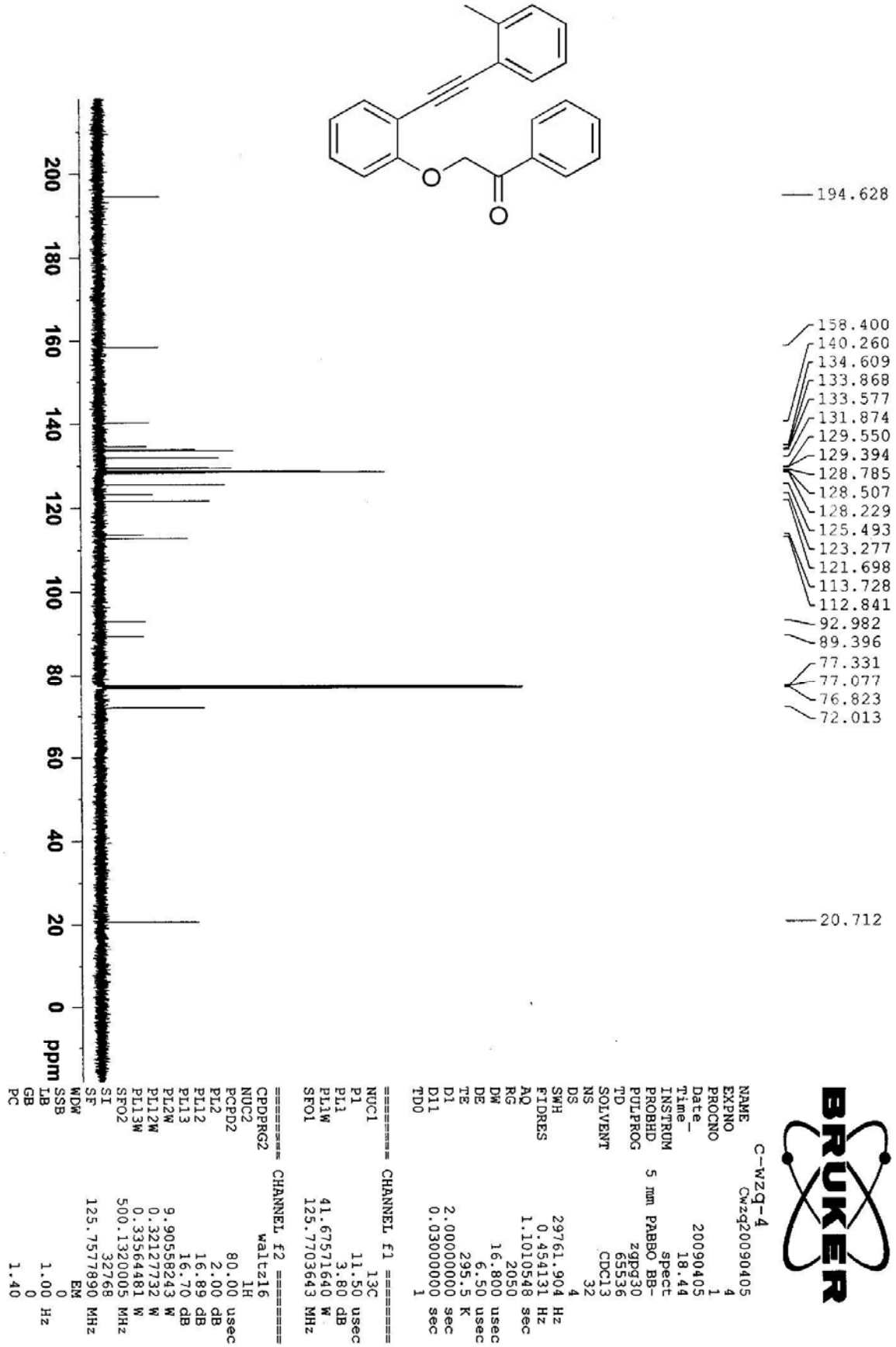
1-Phenyl-2-(2-(2-*m*-tolylethynyl)phenoxy)ethanone (1b)



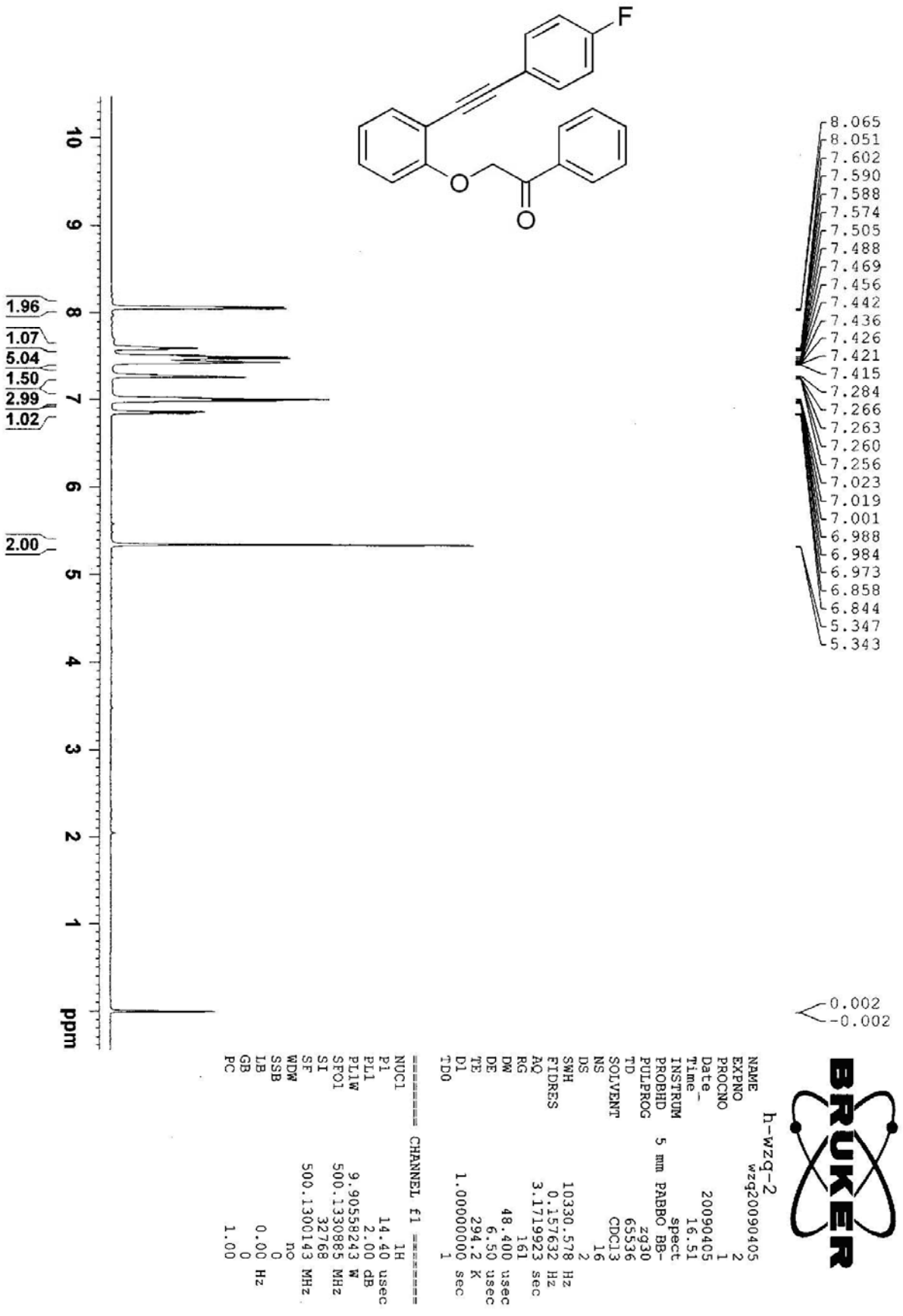
1-Phenyl-2-(2-(2-*o*-tolylethynyl)phenoxy)ethanone (1c)



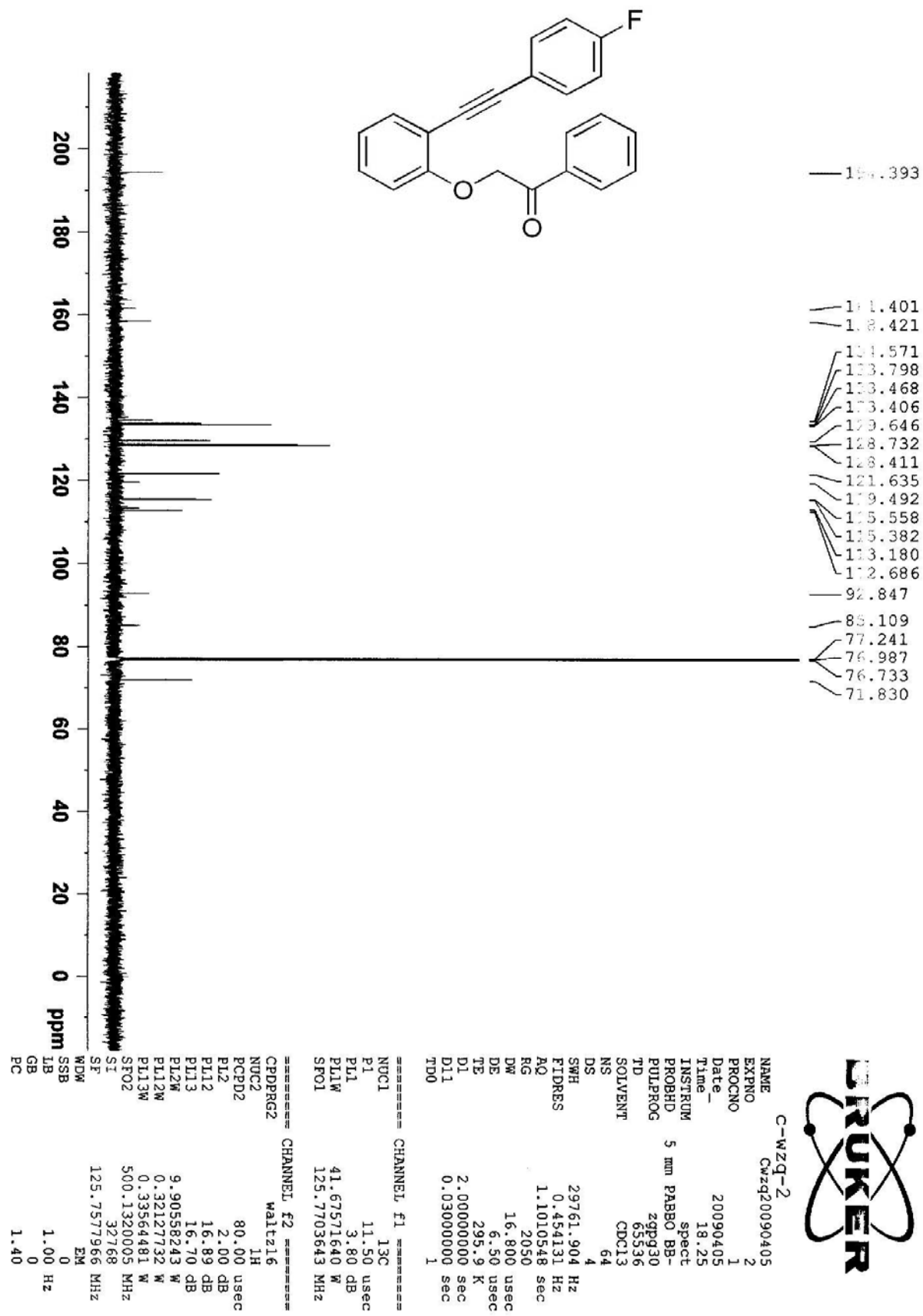
1-Phenyl-2-(2-(2-*o*-tolylethynyl)phenoxy)ethanone (1c)



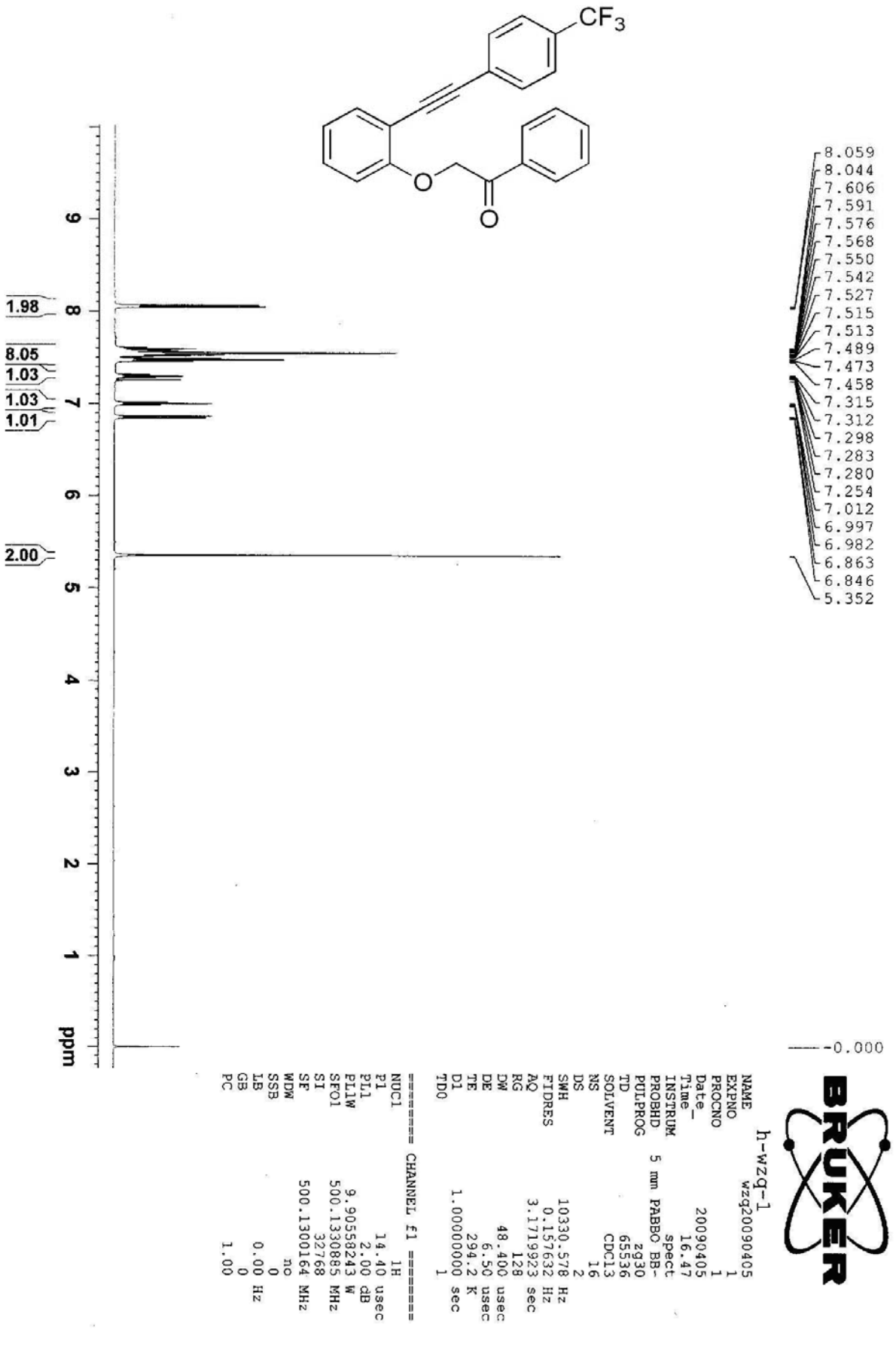
2-(2-(2-(4-Fluorophenyl)ethynyl)phenoxy)-1-phenylethanone (1d)



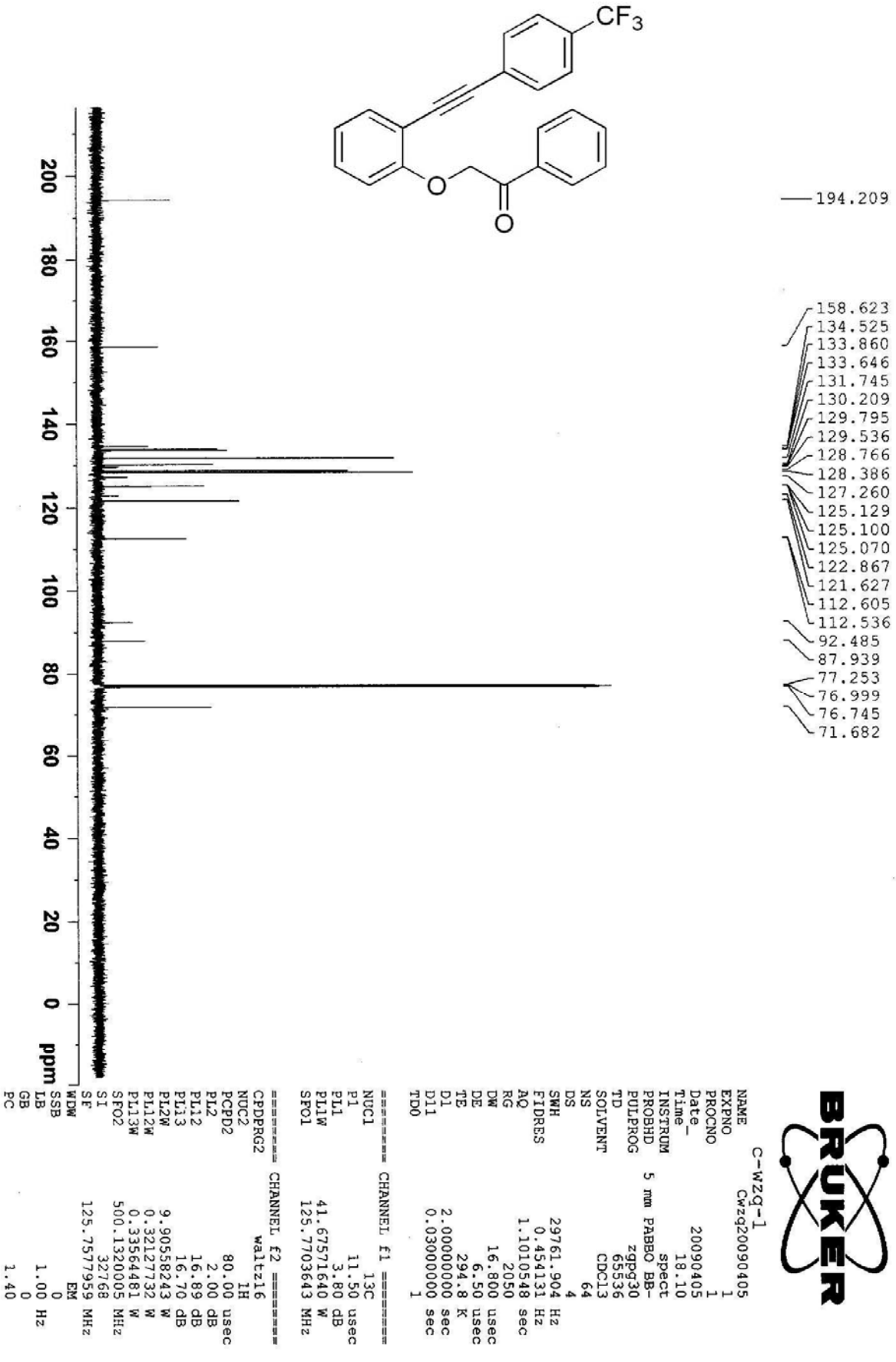
2-(2-(2-(4-Fluorophenyl)ethynyl)phenoxy)-1-phenylethanone (1d)



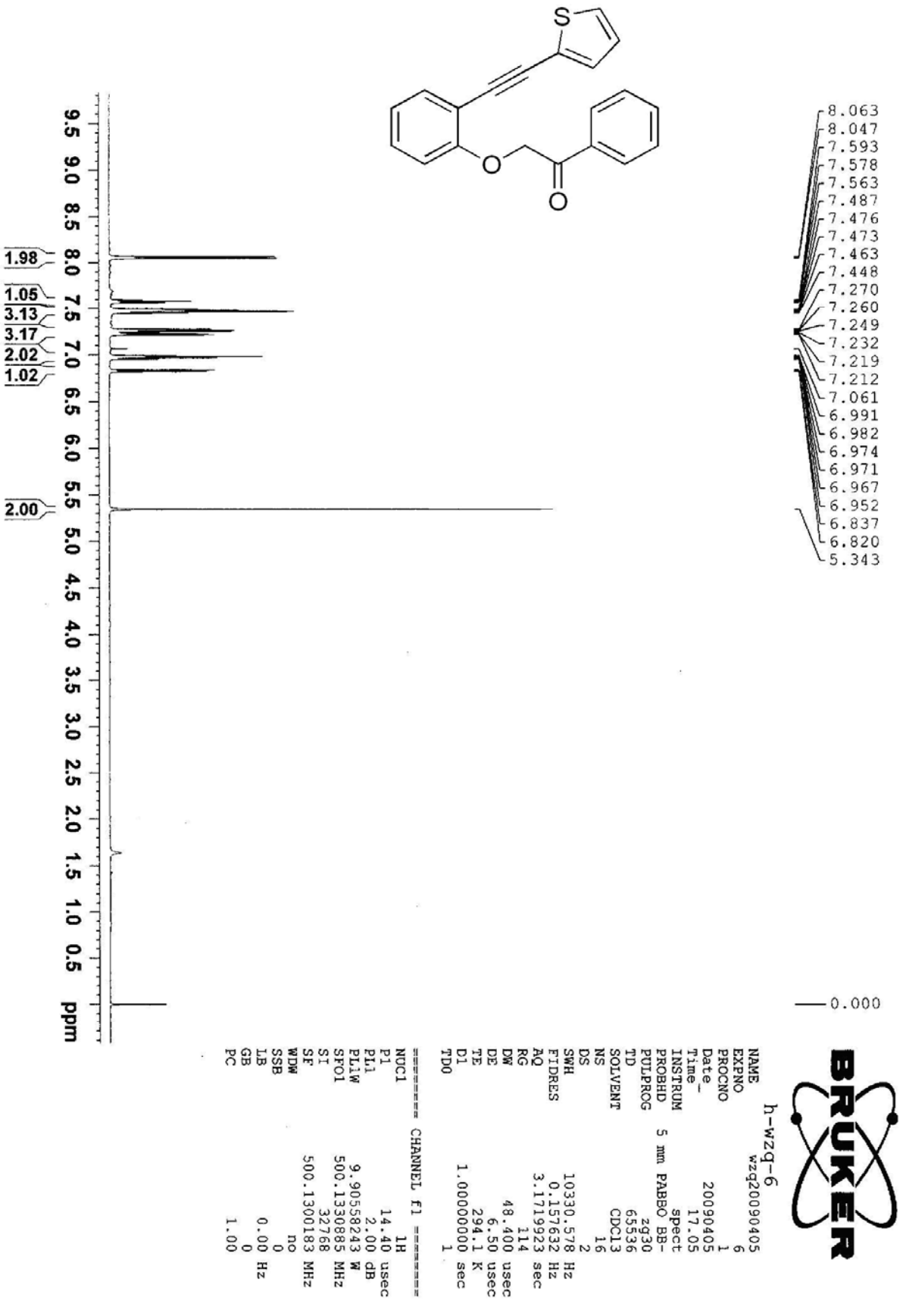
1-Phenyl-2-(2-(2-(4-(trifluoromethyl)phenyl)ethynyl)phenoxy)ethanone (1e)



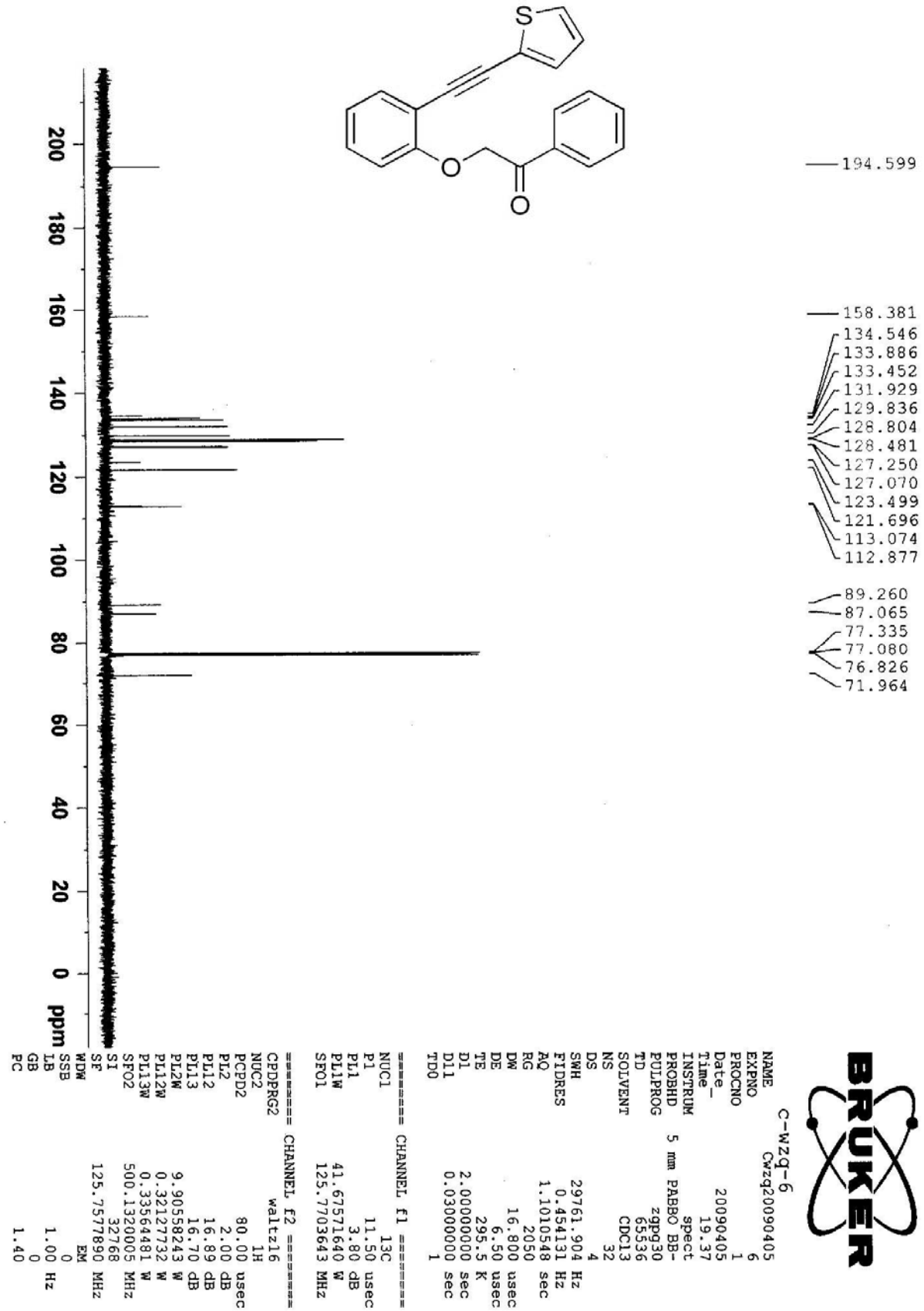
1-Phenyl-2-(2-(2-(4-(trifluoromethyl)phenyl)ethynyl)phenoxy)ethanone (1e)



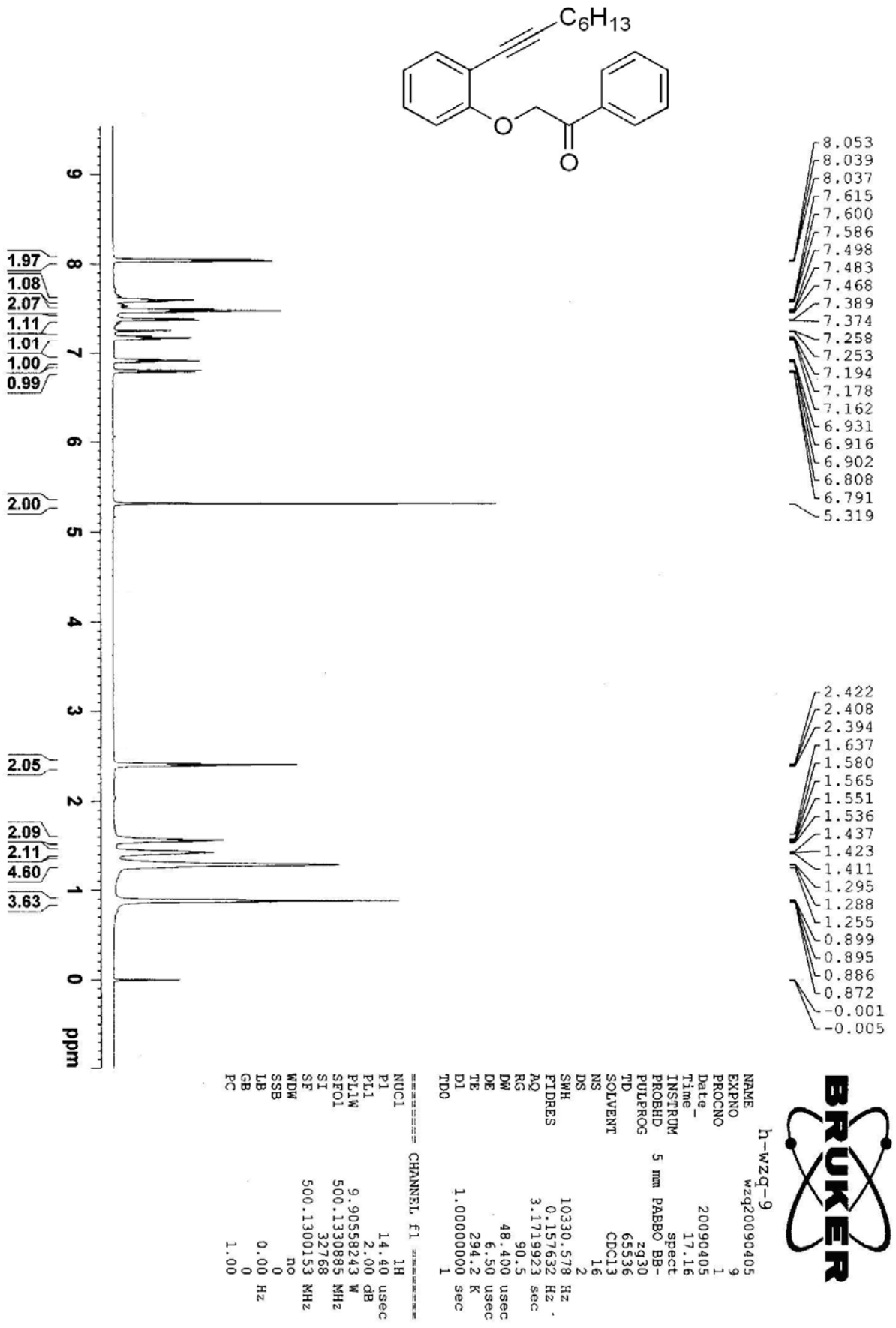
1-Phenyl-2-(2-(2-(thiophen-2-yl)ethynyl)phenoxy)ethanone (1f)



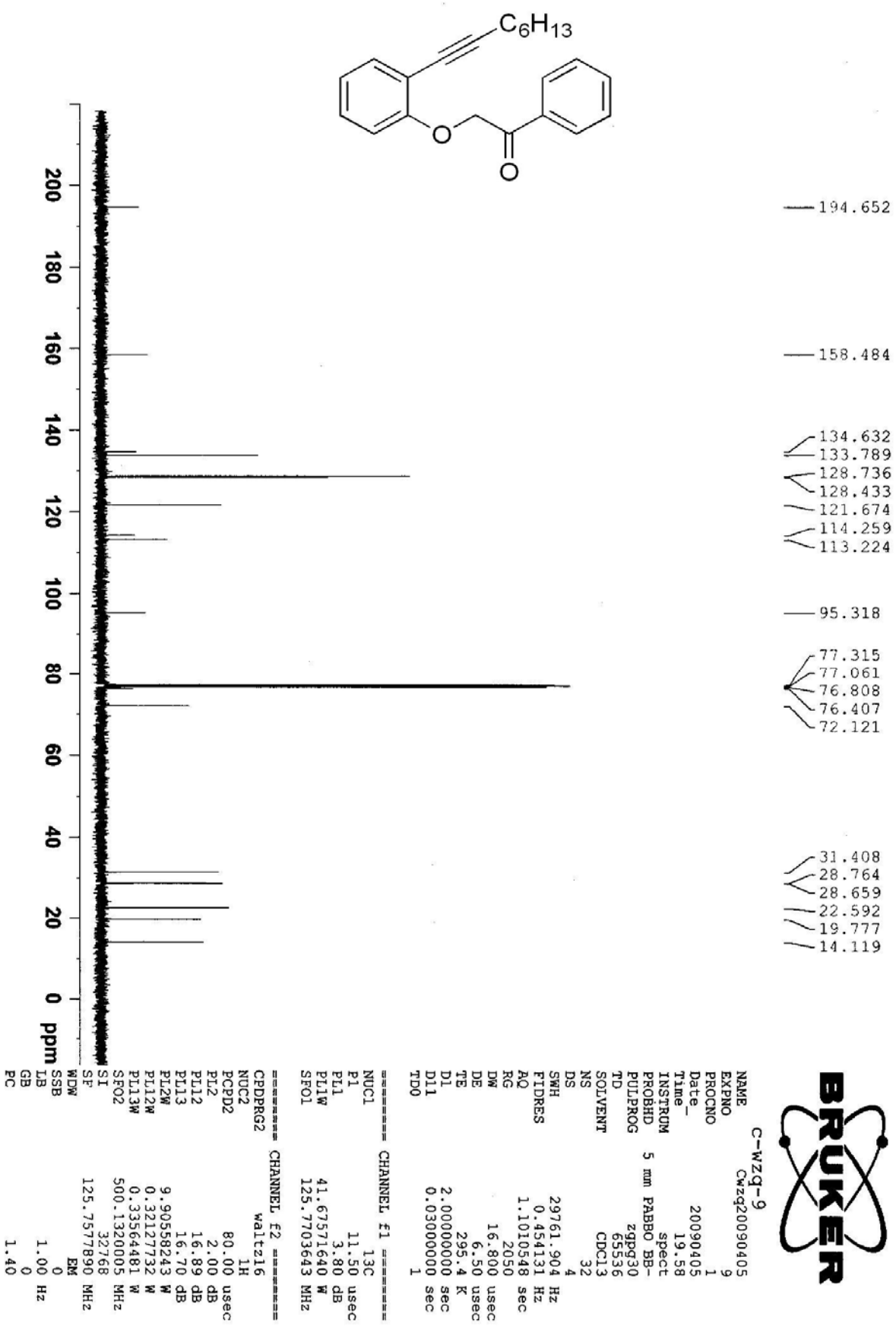
1-Phenyl-2-(2-(2-(thiophen-2-yl)ethynyl)phenoxy)ethanone (1f)



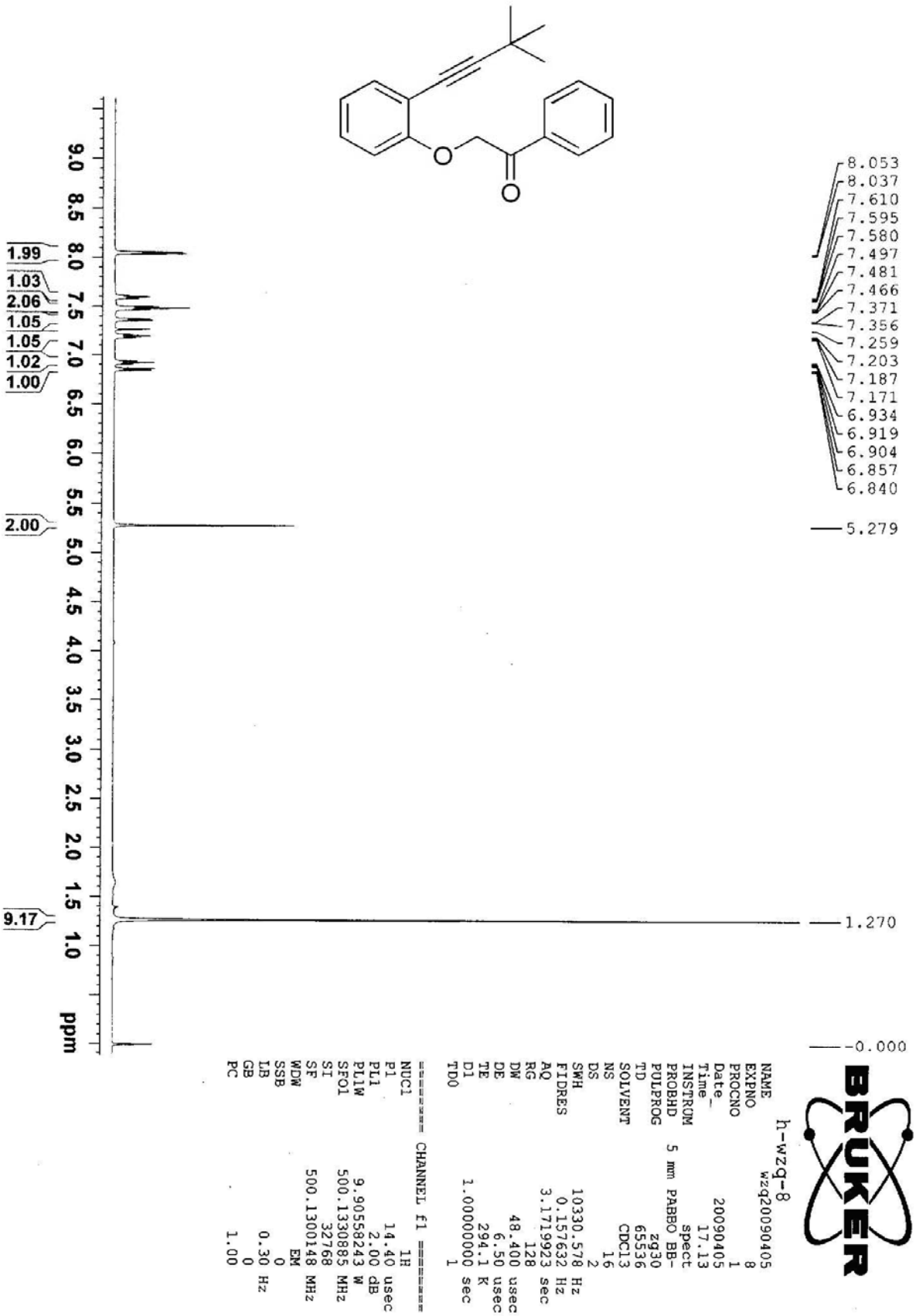
2-(2-(Oct-1-ynyl)phenoxy)-1-phenylethanone (1g)



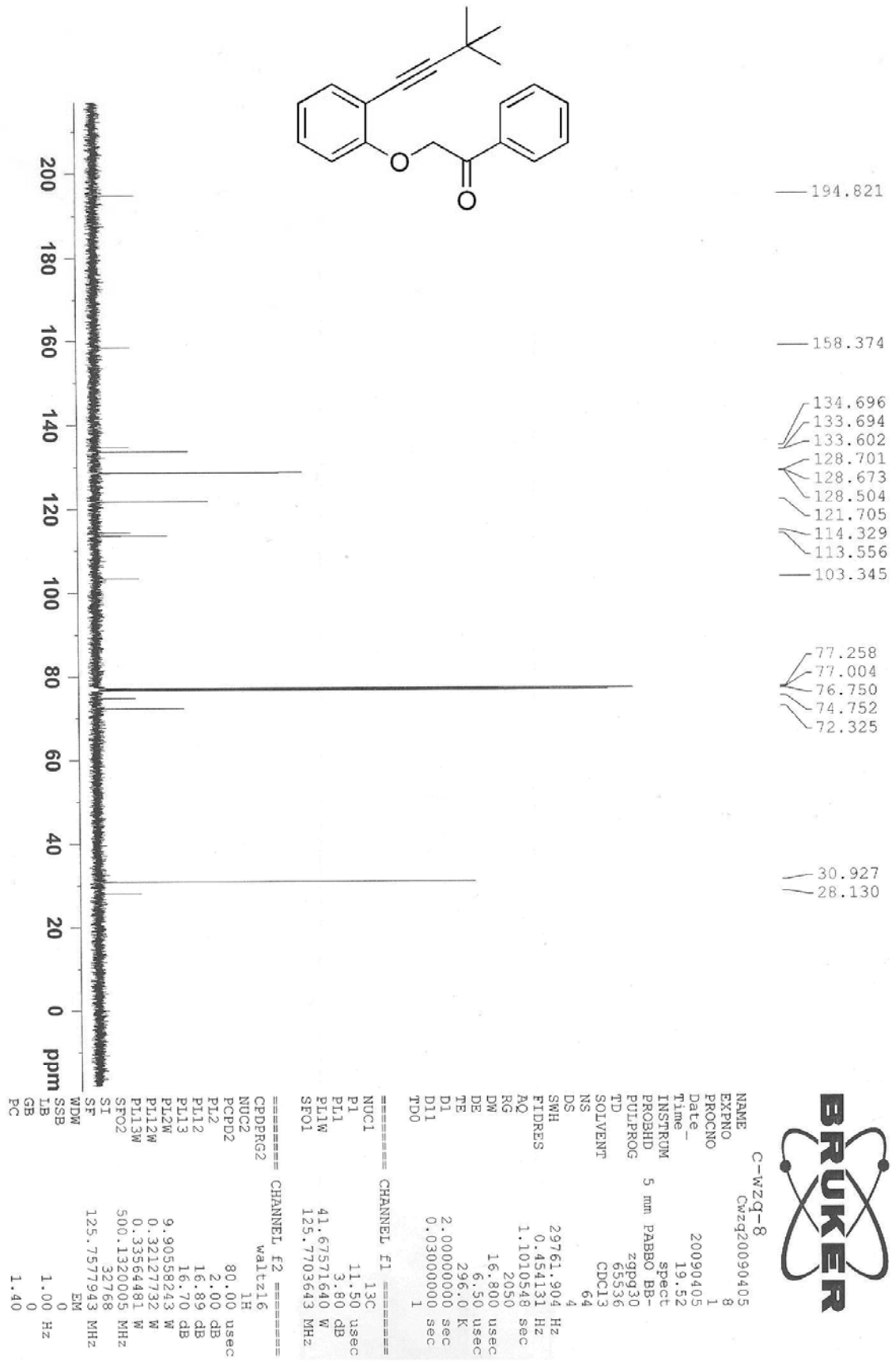
2-(2-(Oct-1-ynyl)phenoxy)-1-phenylethanone (1g)



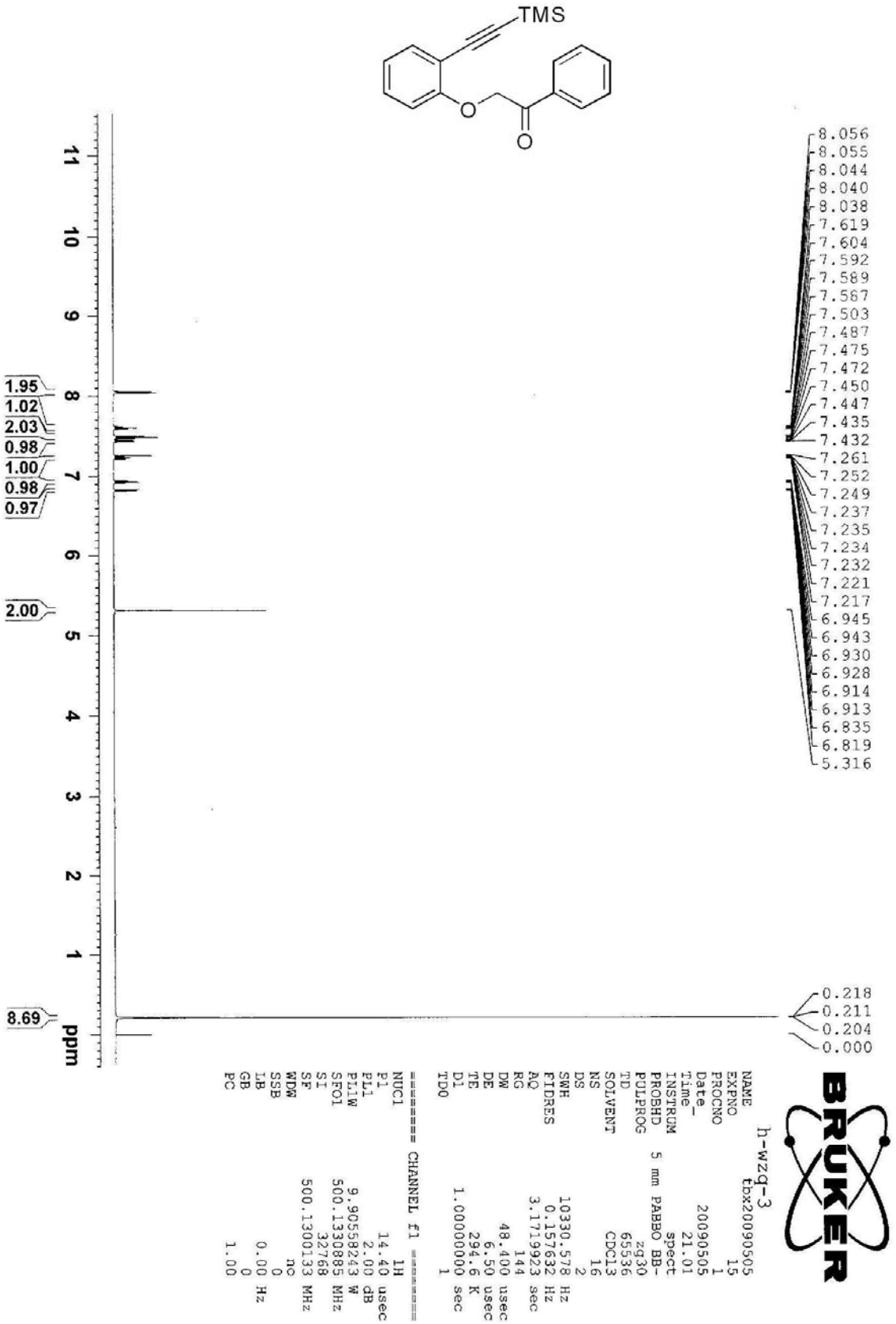
2-(2-(3,3-Dimethylbut-1-ynyl)phenoxy)-1-phenylethanone (1h)



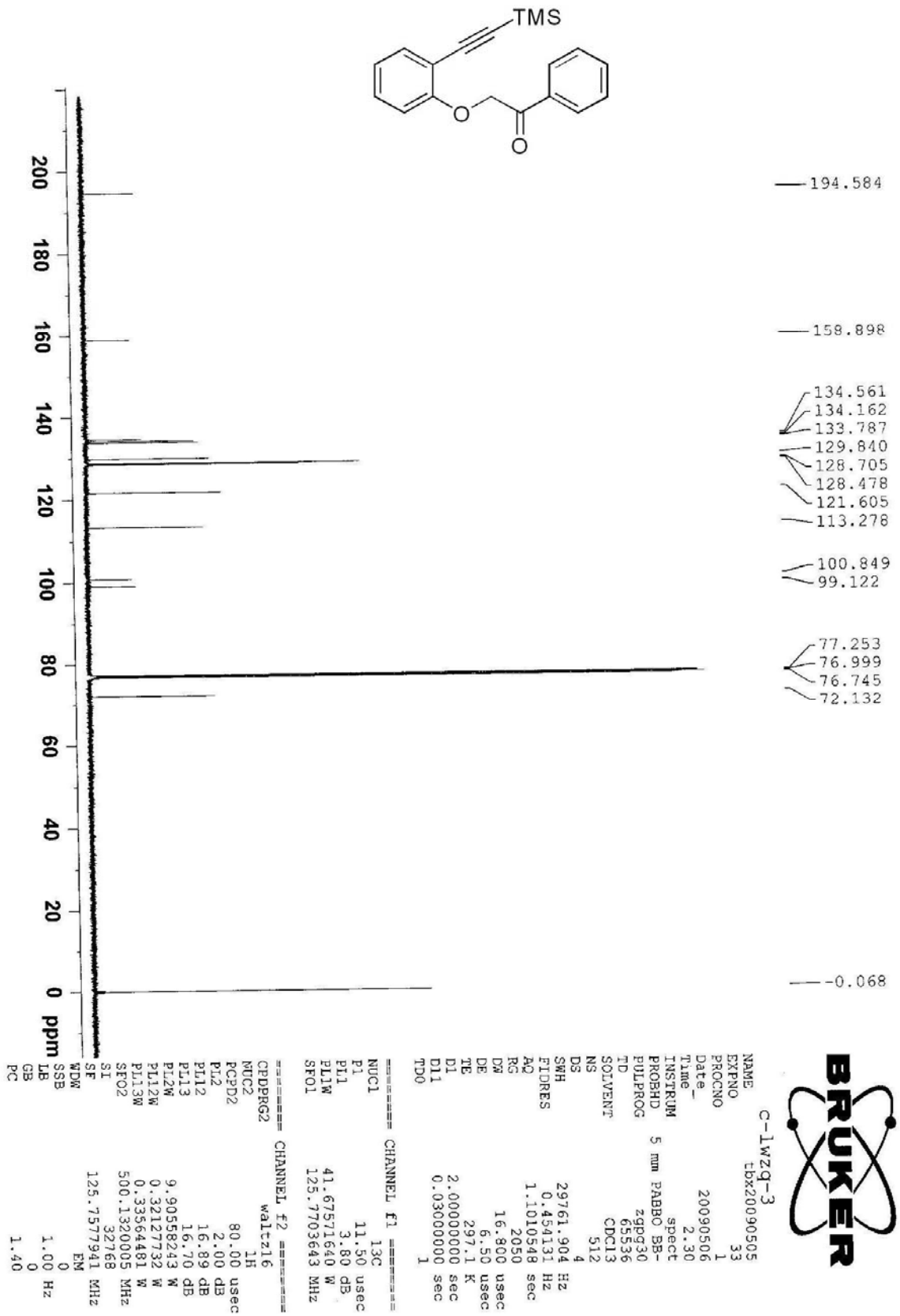
2-(2-(3,3-Dimethylbut-1-ynyl)phenoxy)-1-phenylethanone (1h)



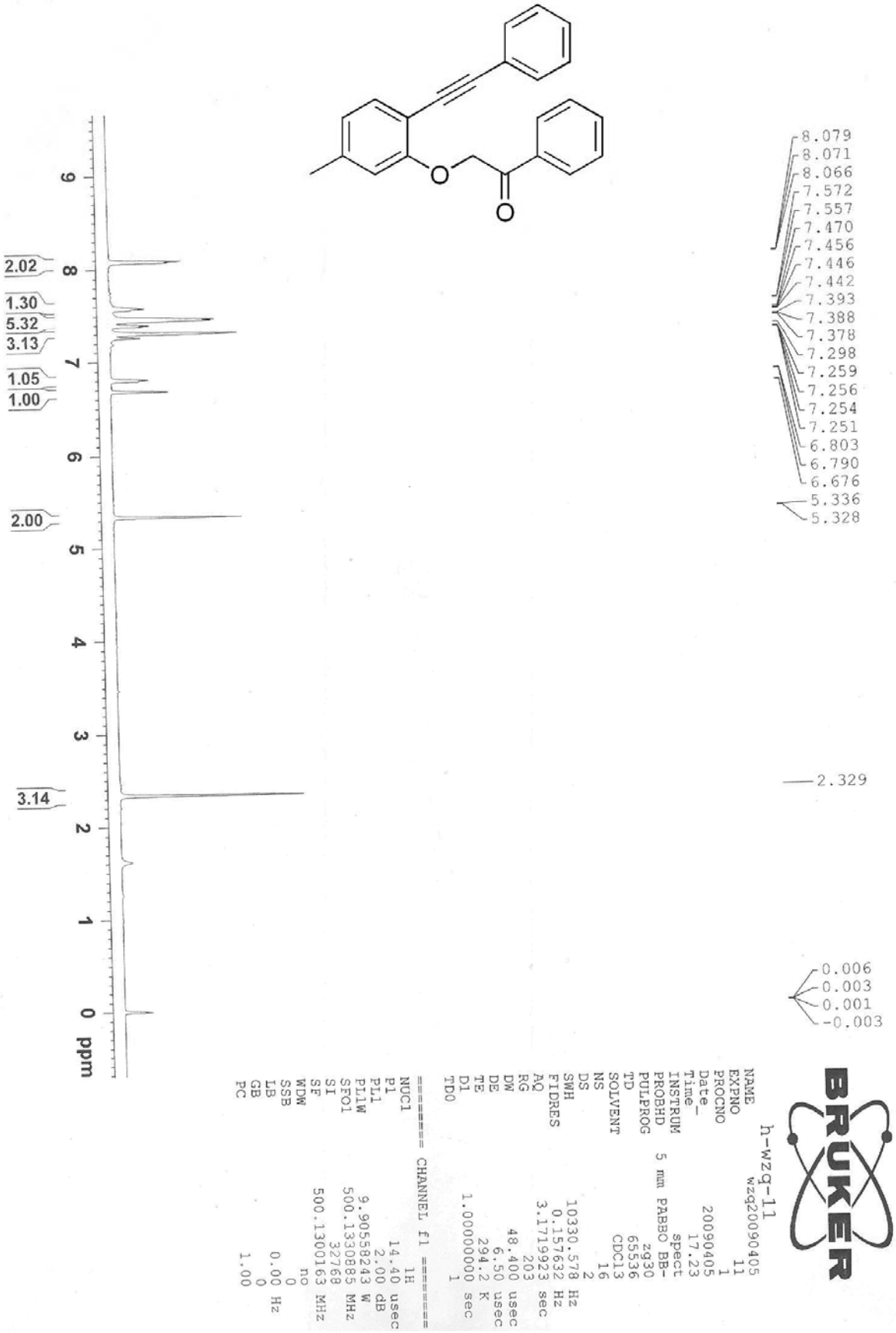
1-Phenyl-2-(2-(2-(trimethylsilyl)ethynyl)phenoxy)ethanone (1i)



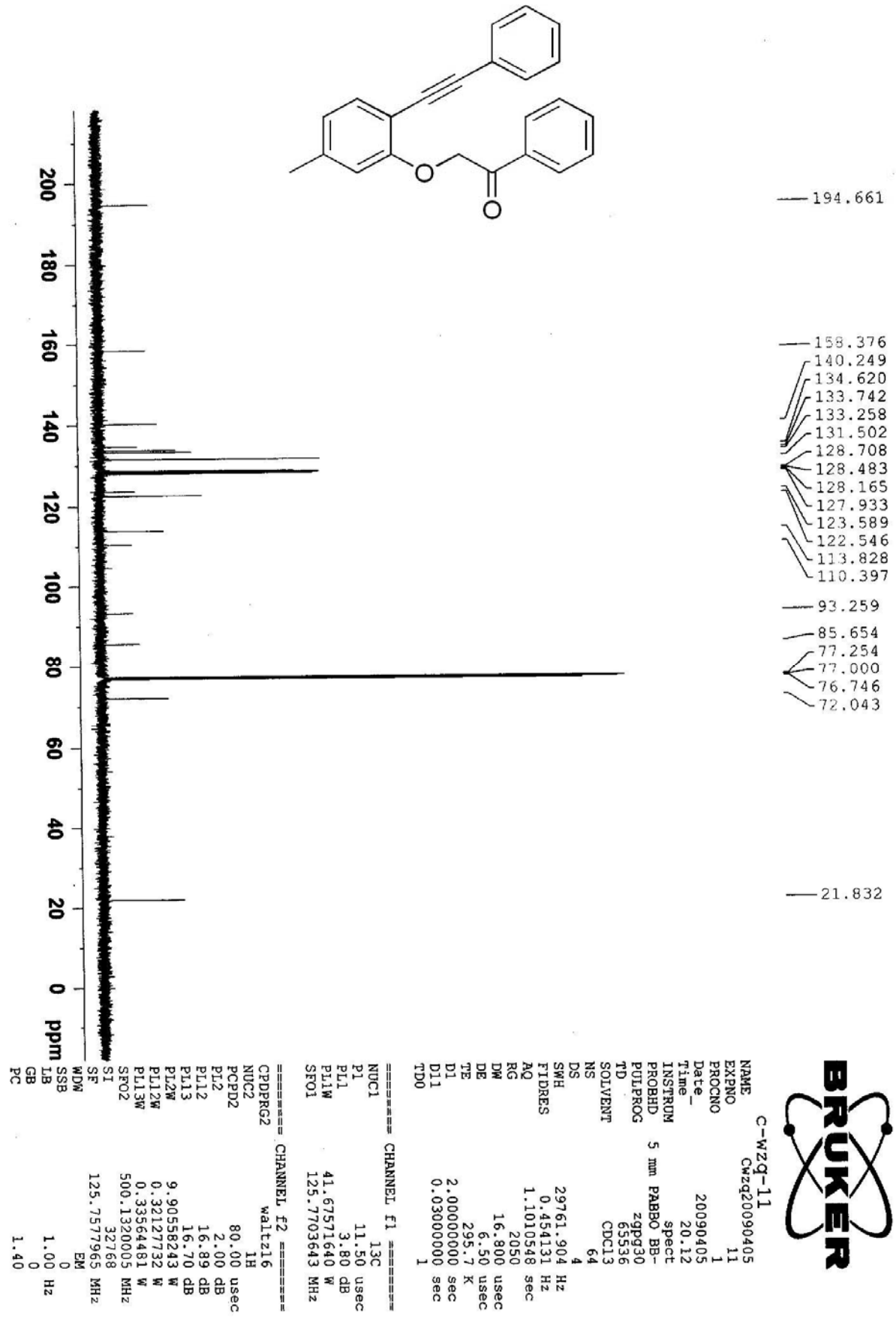
1-Phenyl-2-(2-(2-(trimethylsilyl)ethynyl)phenoxy)ethanone (1i)



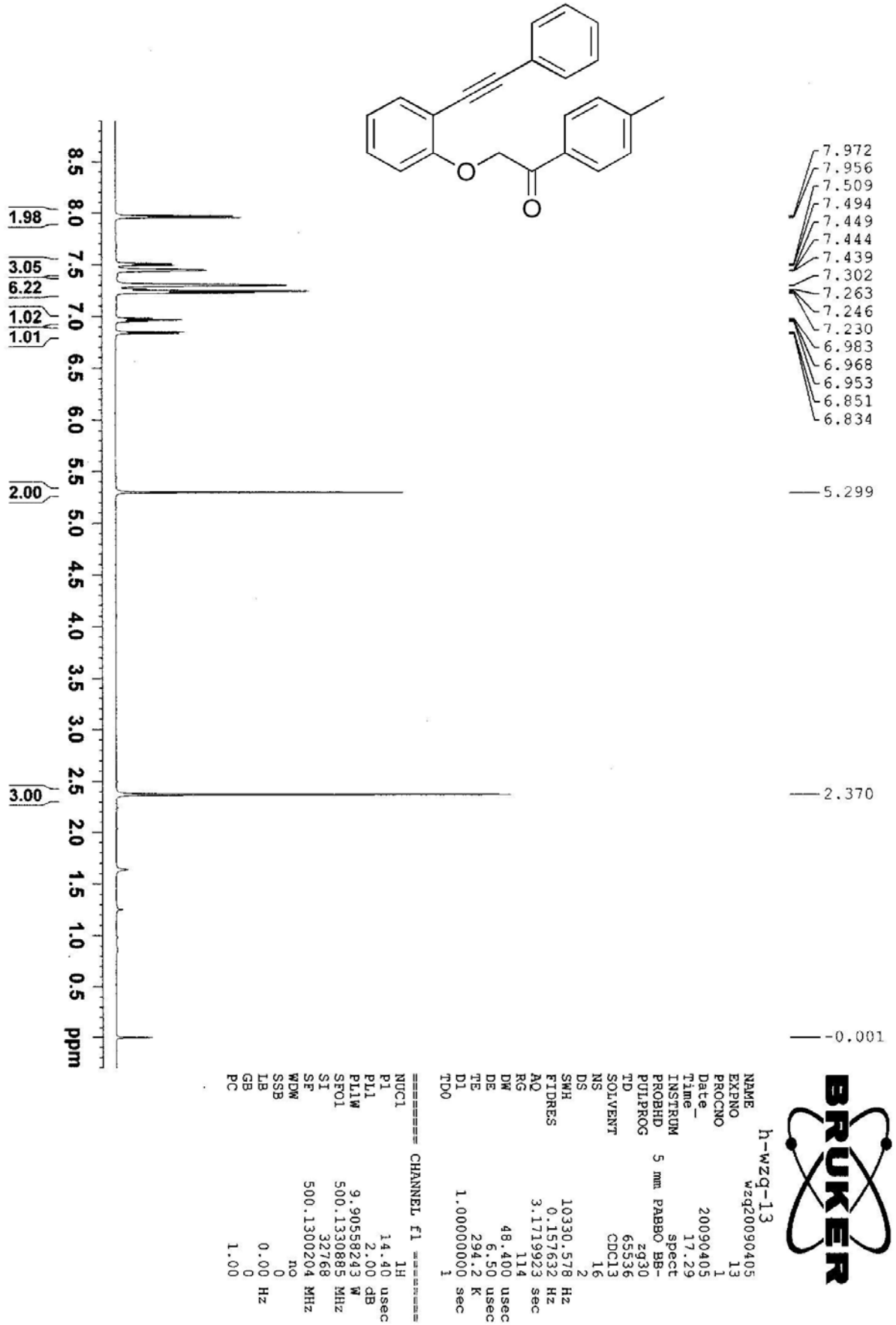
2-(5-Methyl-2-(2-phenylethynyl)phenoxy)-1-phenylethanone (1j)



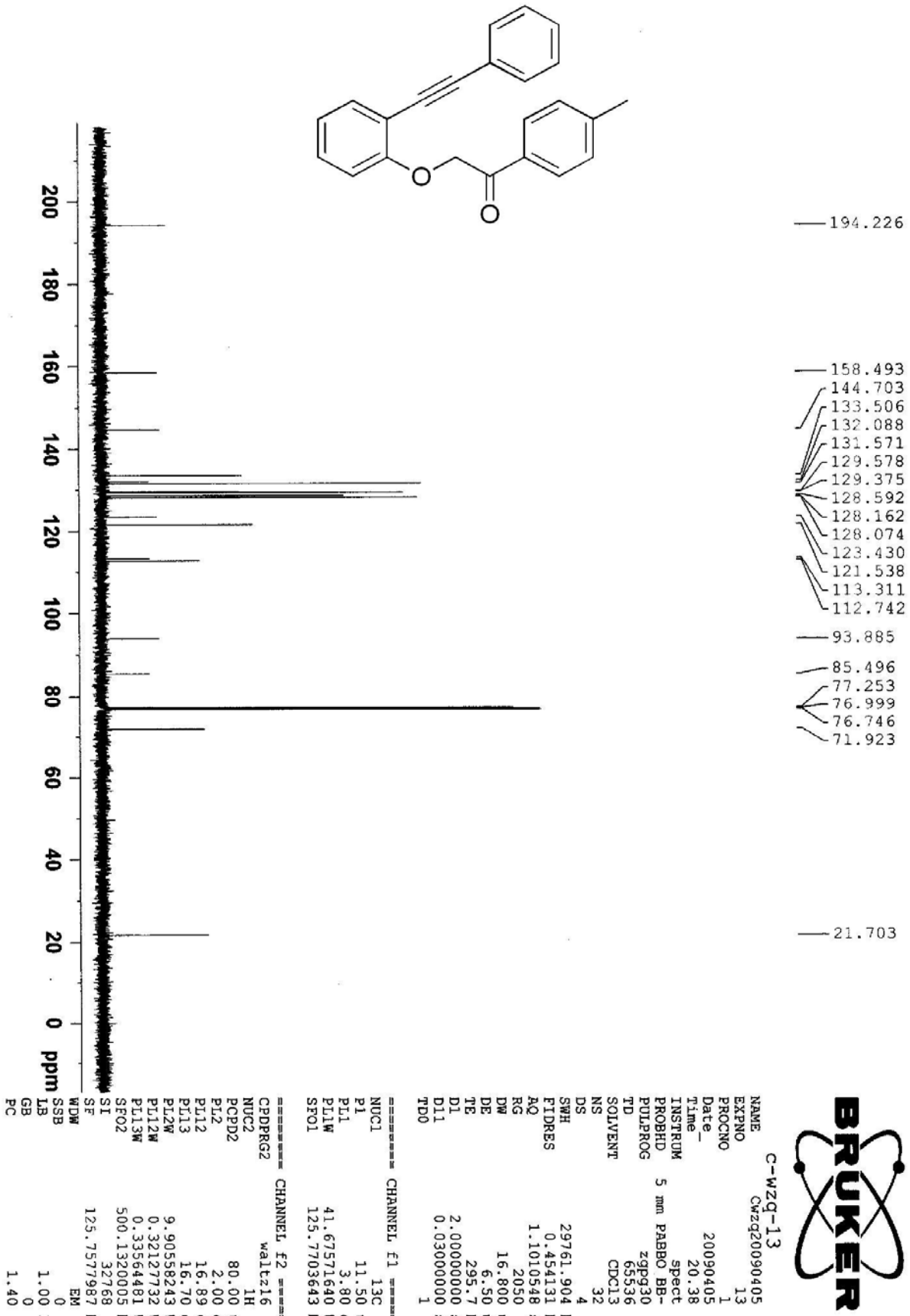
2-(5-Methyl-2-(2-phenylethynyl)phenoxy)-1-phenylethanone (1j)



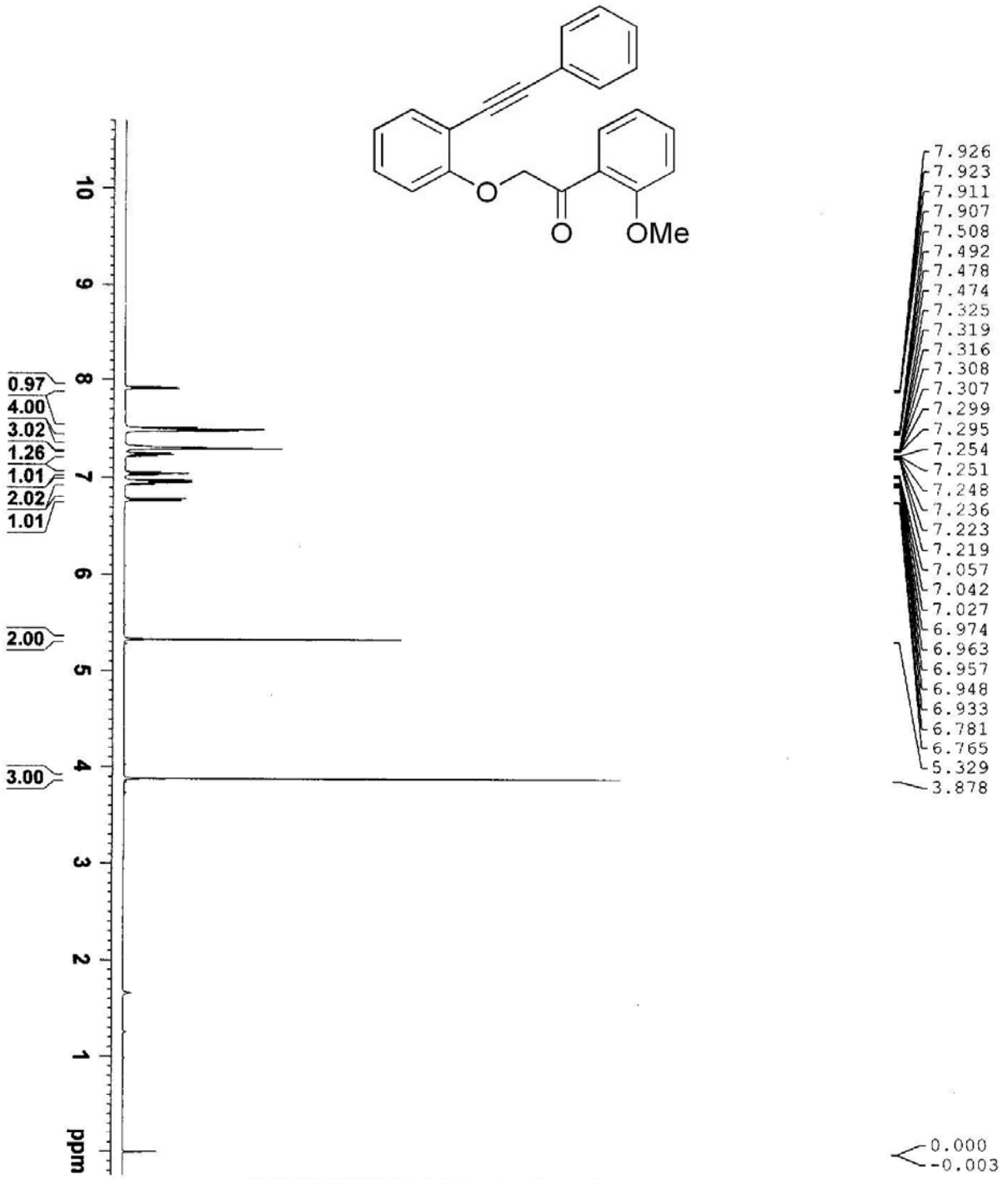
2-(2-(2-Phenylethynyl)phenoxy)-1-p-tolylethanone (1k)



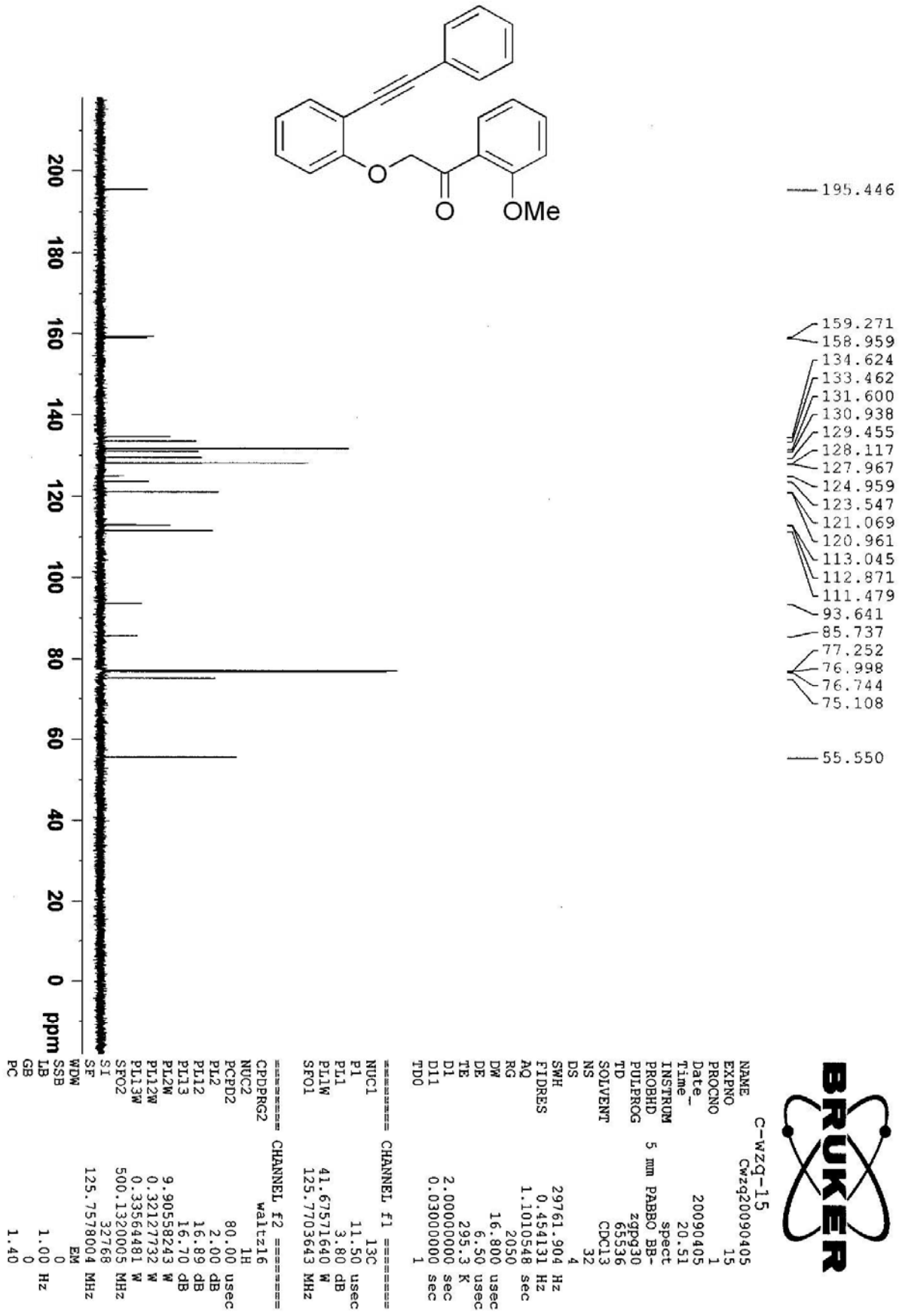
2-(2-(2-Phenylethynyl)phenoxy)-1-*p*-tolylethanone (1k)



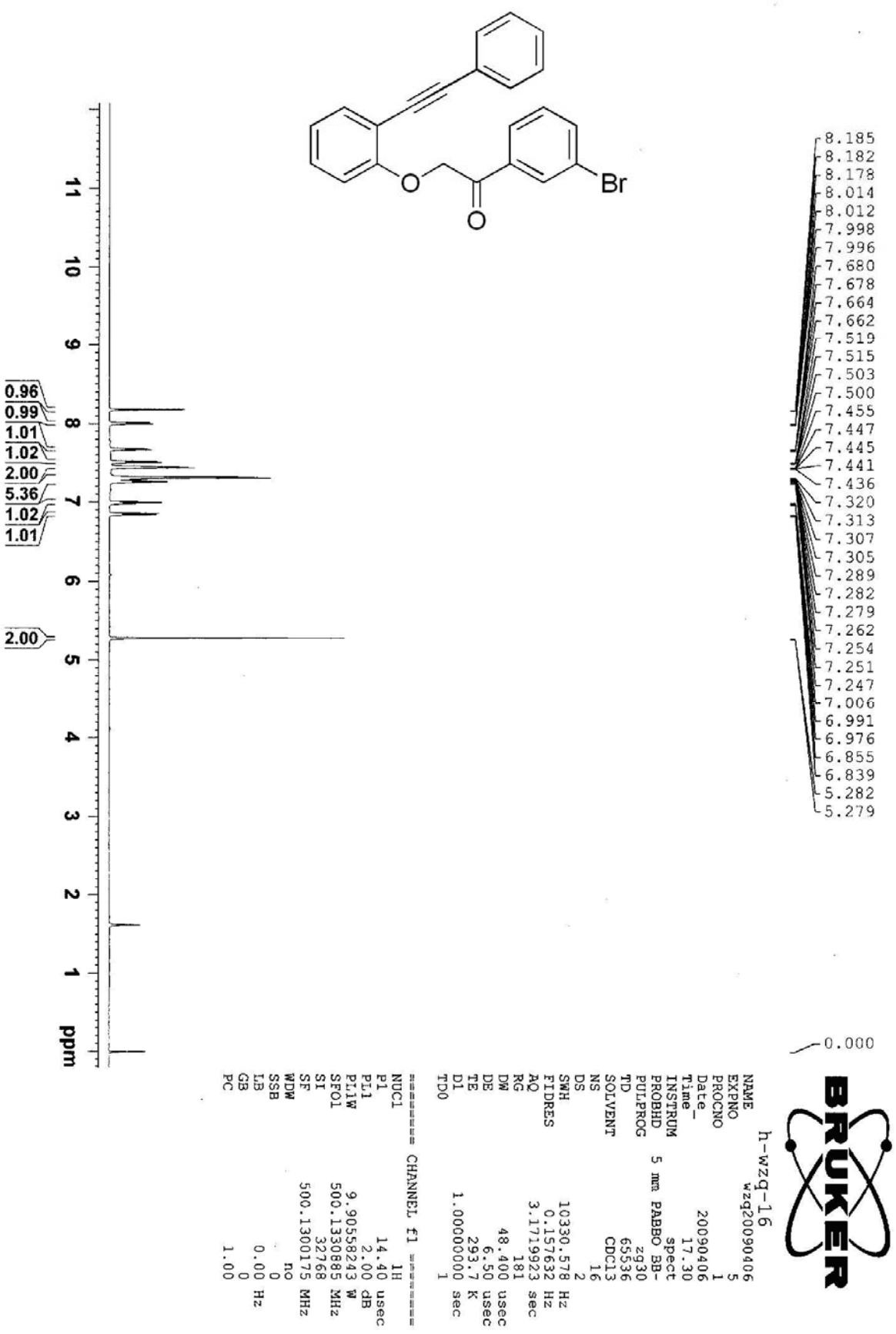
1-(2-Methoxyphenyl)-2-(2-(2-phenylethynyl)phenoxy)ethanone (1l)



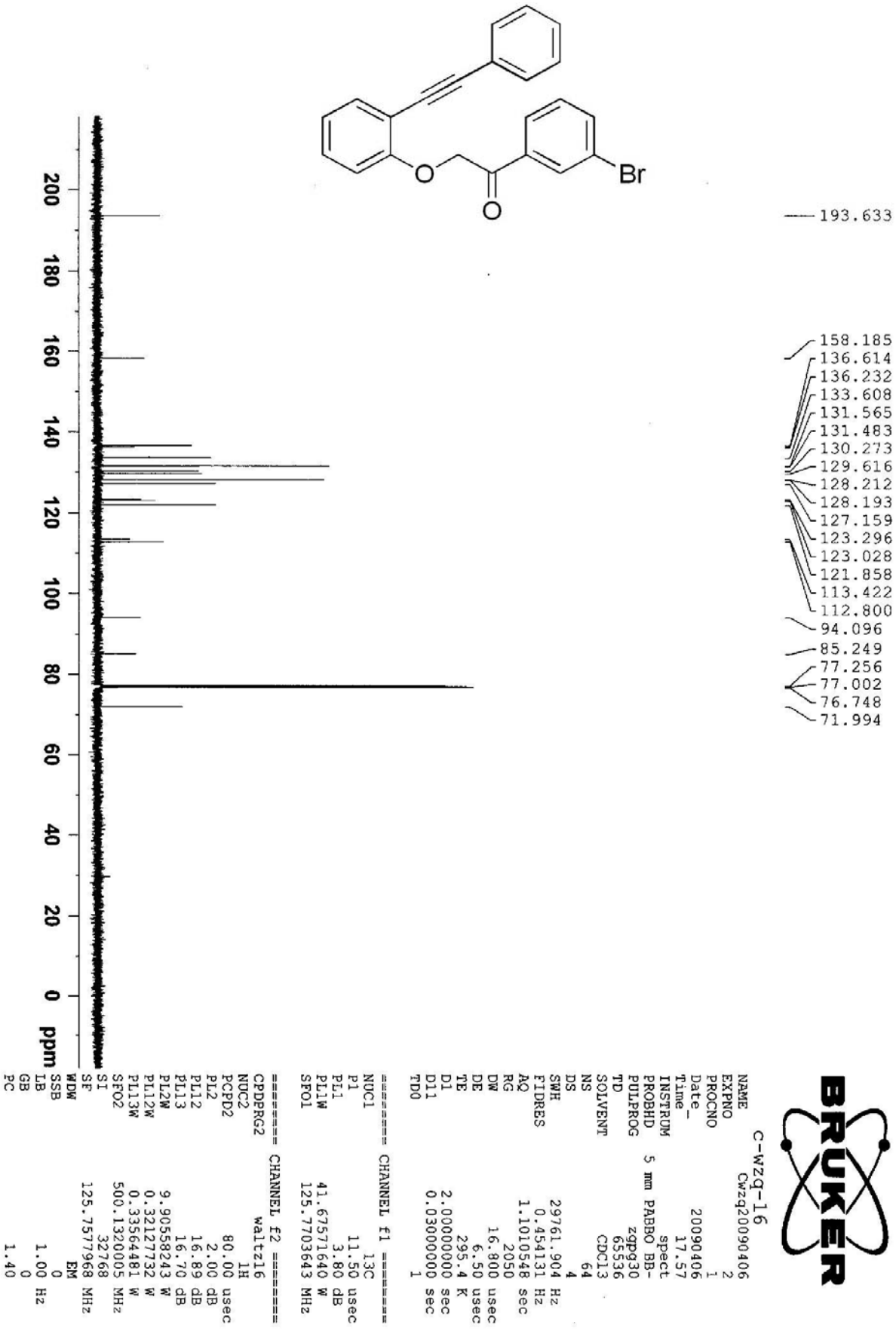
1-(2-Methoxyphenyl)-2-(2-(2-phenylethynyl)phenoxy)ethanone (1l)



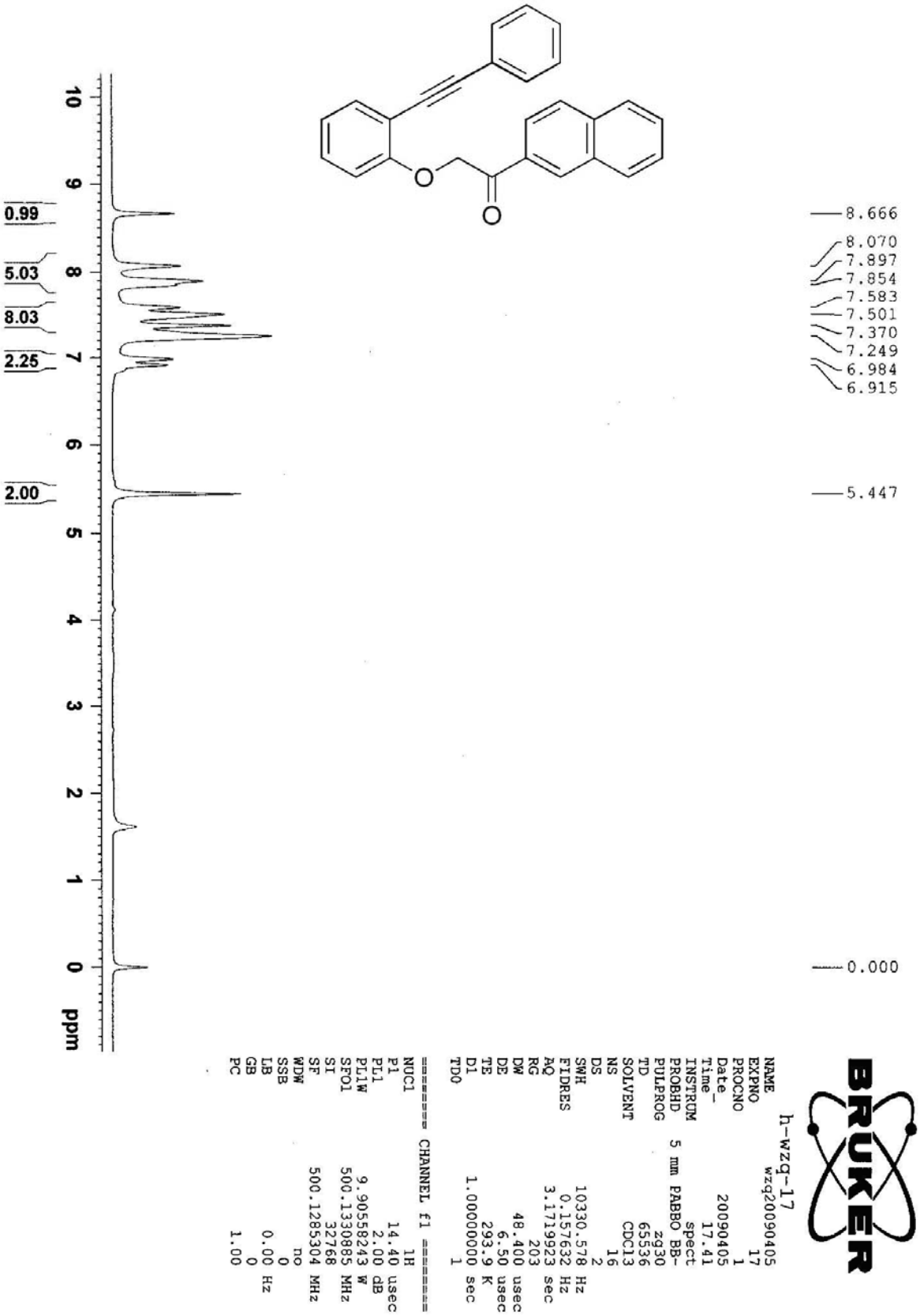
1-(3-Bromophenyl)-2-(2-(2-phenylethynyl)phenoxy)ethanone (1m)



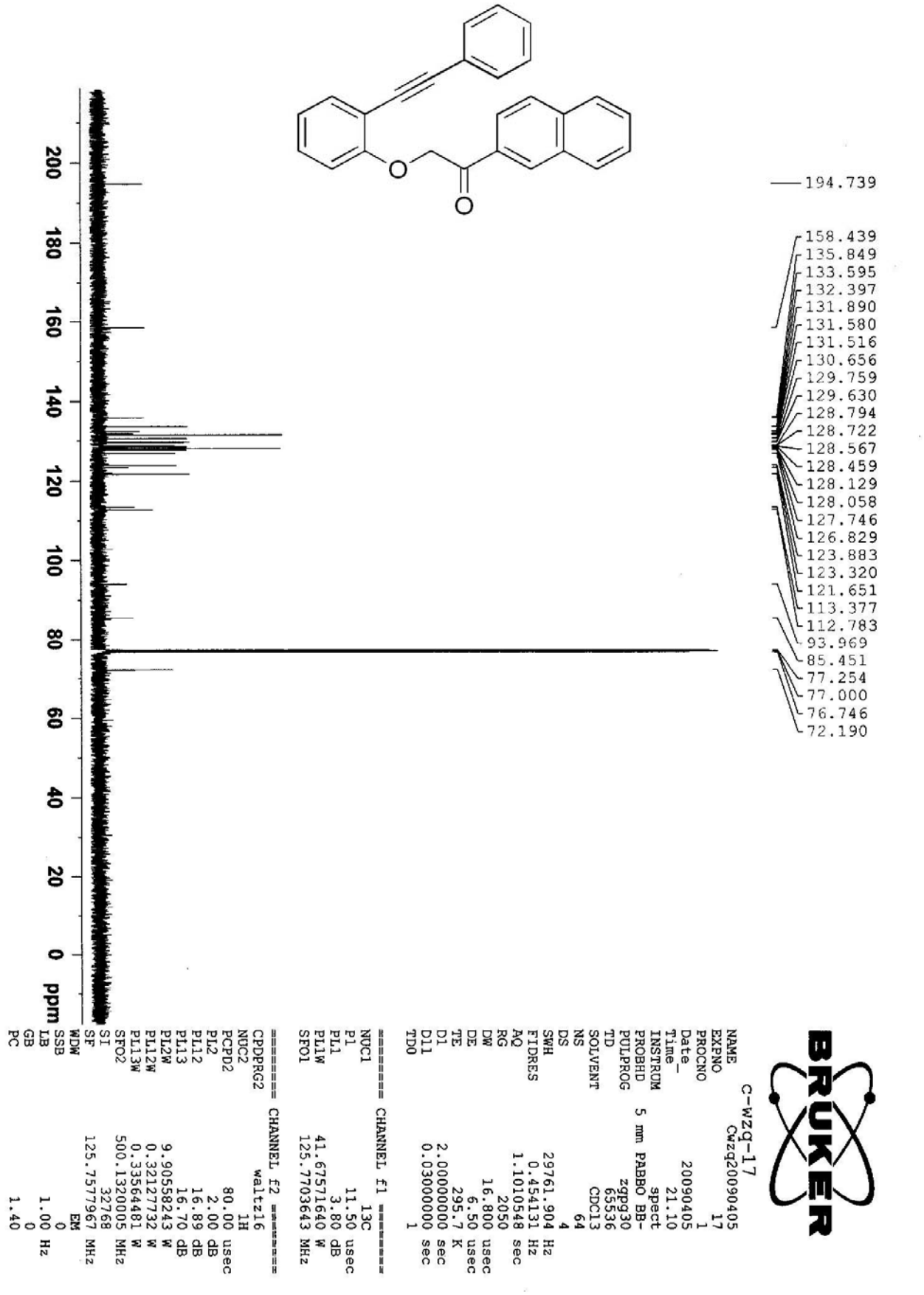
1-(3-Bromophenyl)-2-(2-(2-phenylethynyl)phenoxy)ethanone (1m)



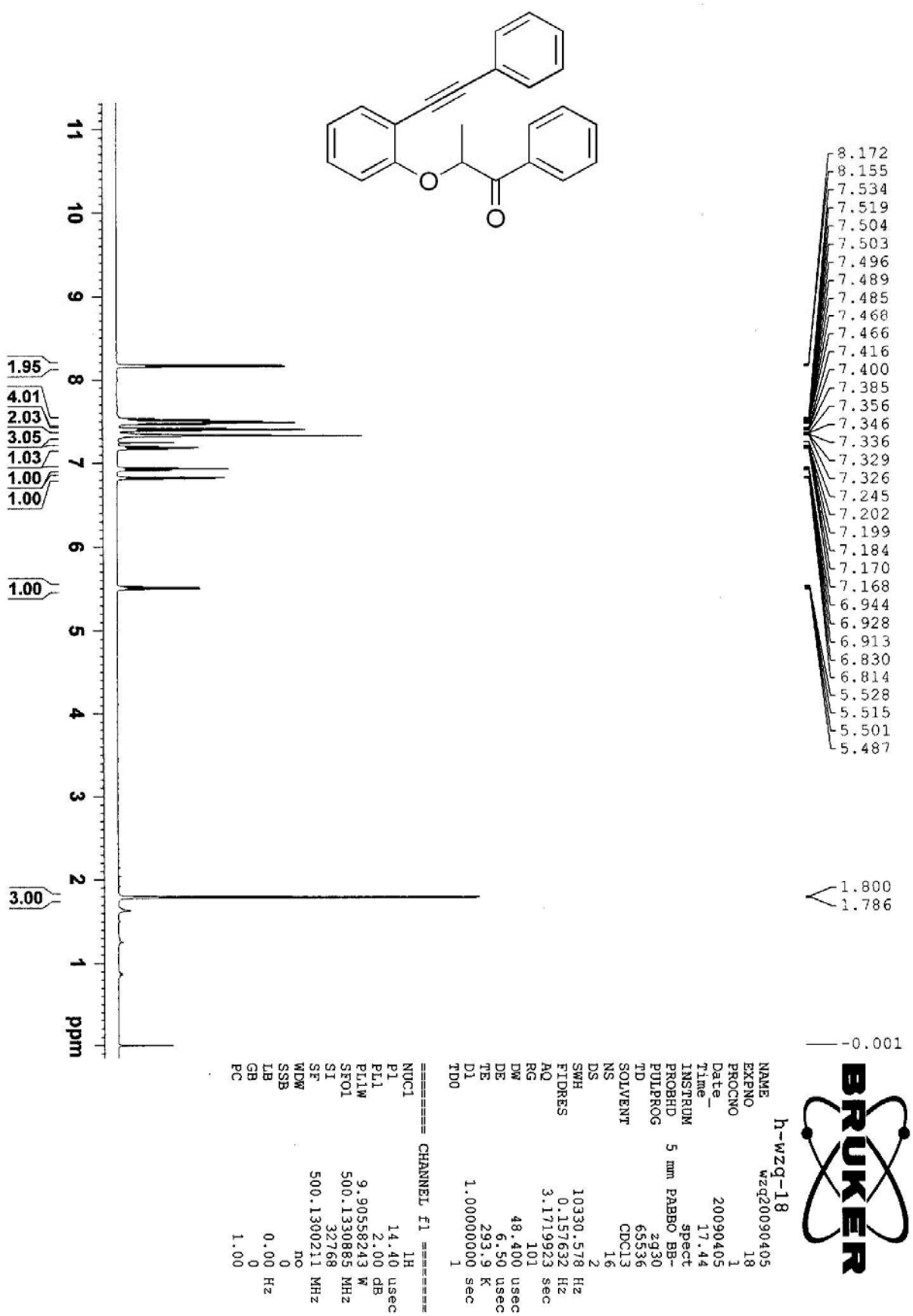
1-(Naphthalen-2-yl)-2-(2-(2-phenylethynyl)phenoxy)ethanone (1n)



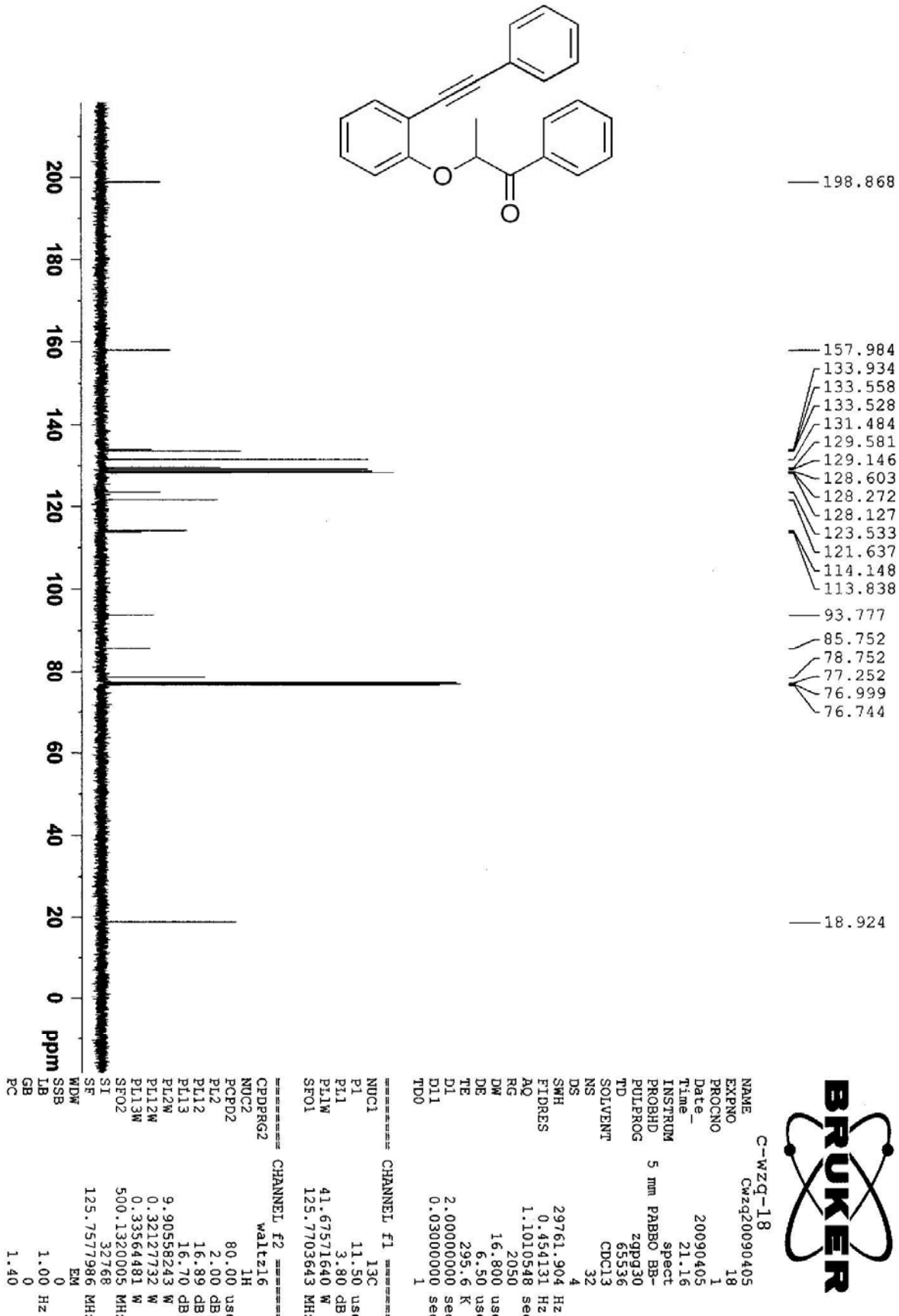
1-(Naphthalen-2-yl)-2-(2-(2-phenylethynyl)phenoxy)ethanone (1n)



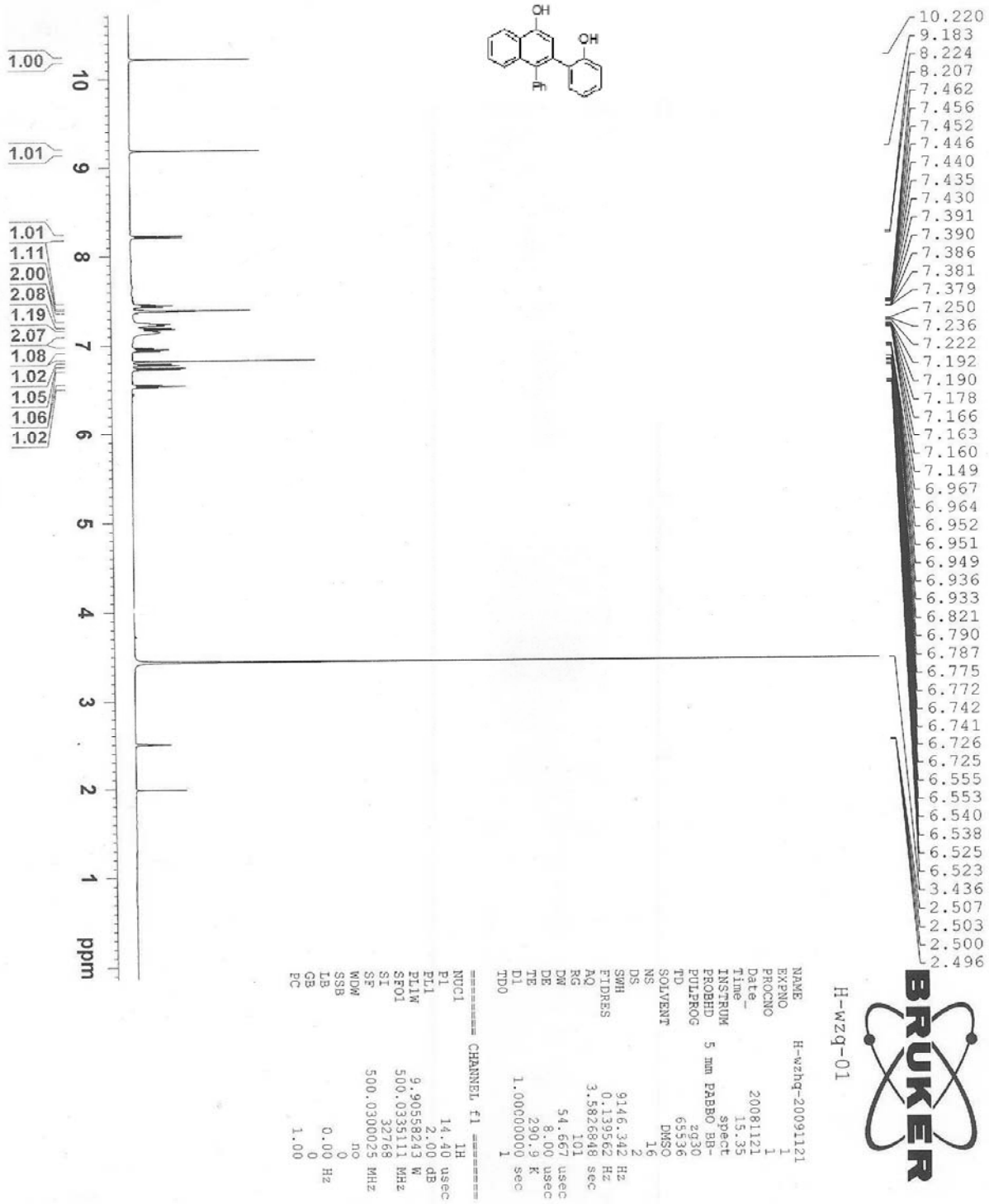
1-Phenyl-2-(2-(2-phenylethynyl)phenoxy)propan-1-one (1o)



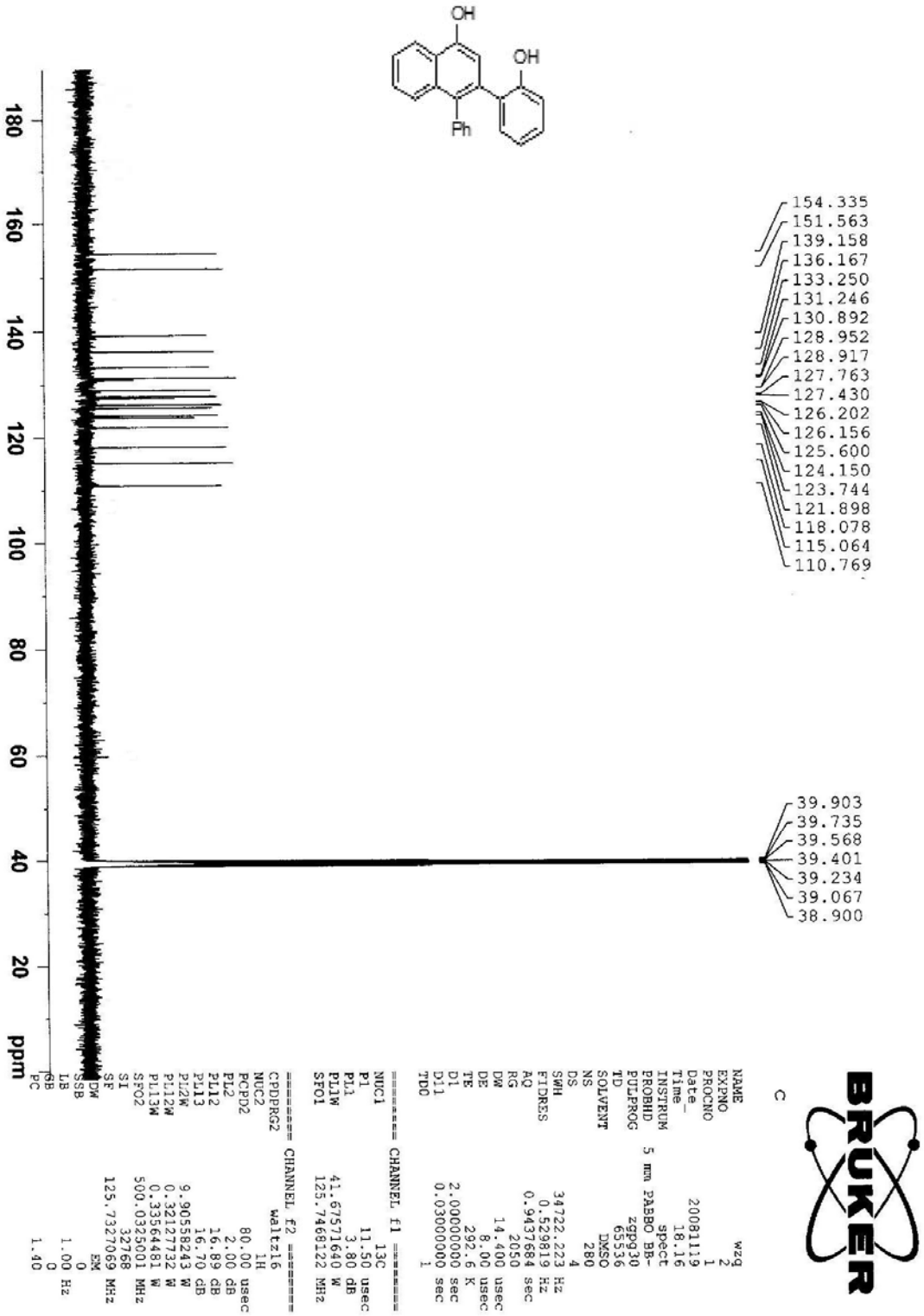
1-Phenyl-2-(2-(2-phenylethynyl)phenoxy)propan-1-one (1o)



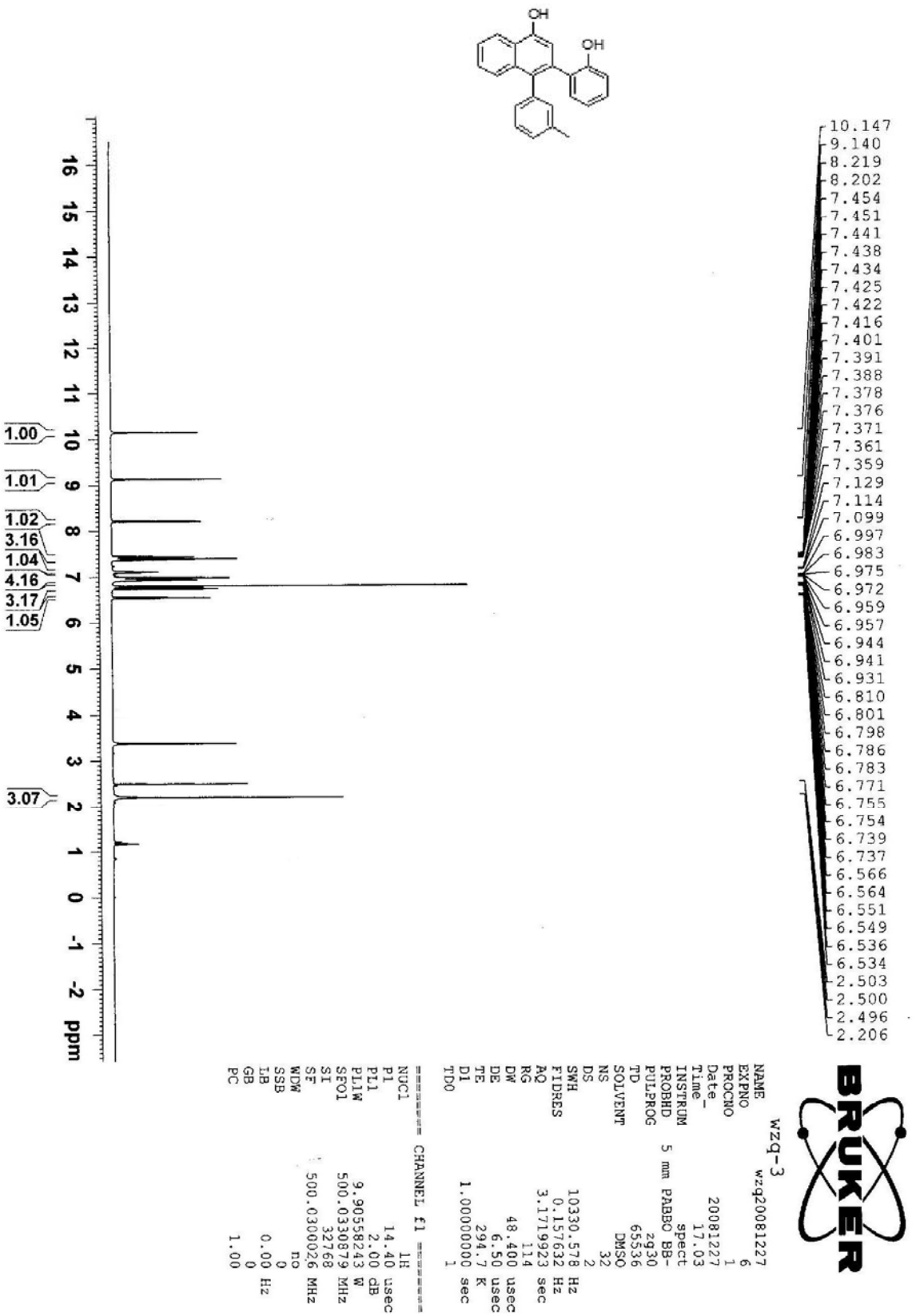
3-(2-Hydroxyphenyl)-4-phenylnaphthalen-1-ol (2a)



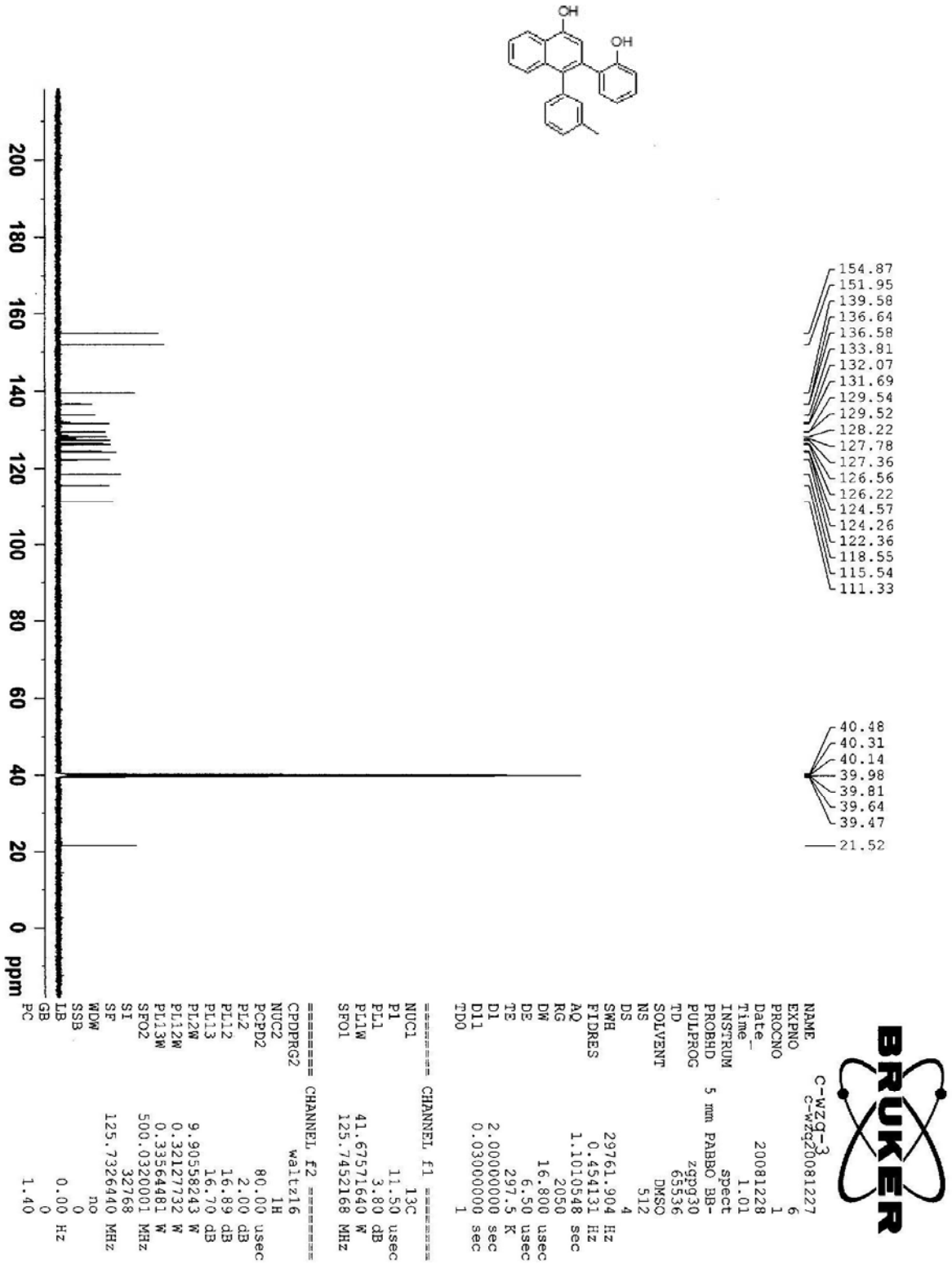
3-(2-Hydroxyphenyl)-4-phenylnaphthalen-1-ol (2a)



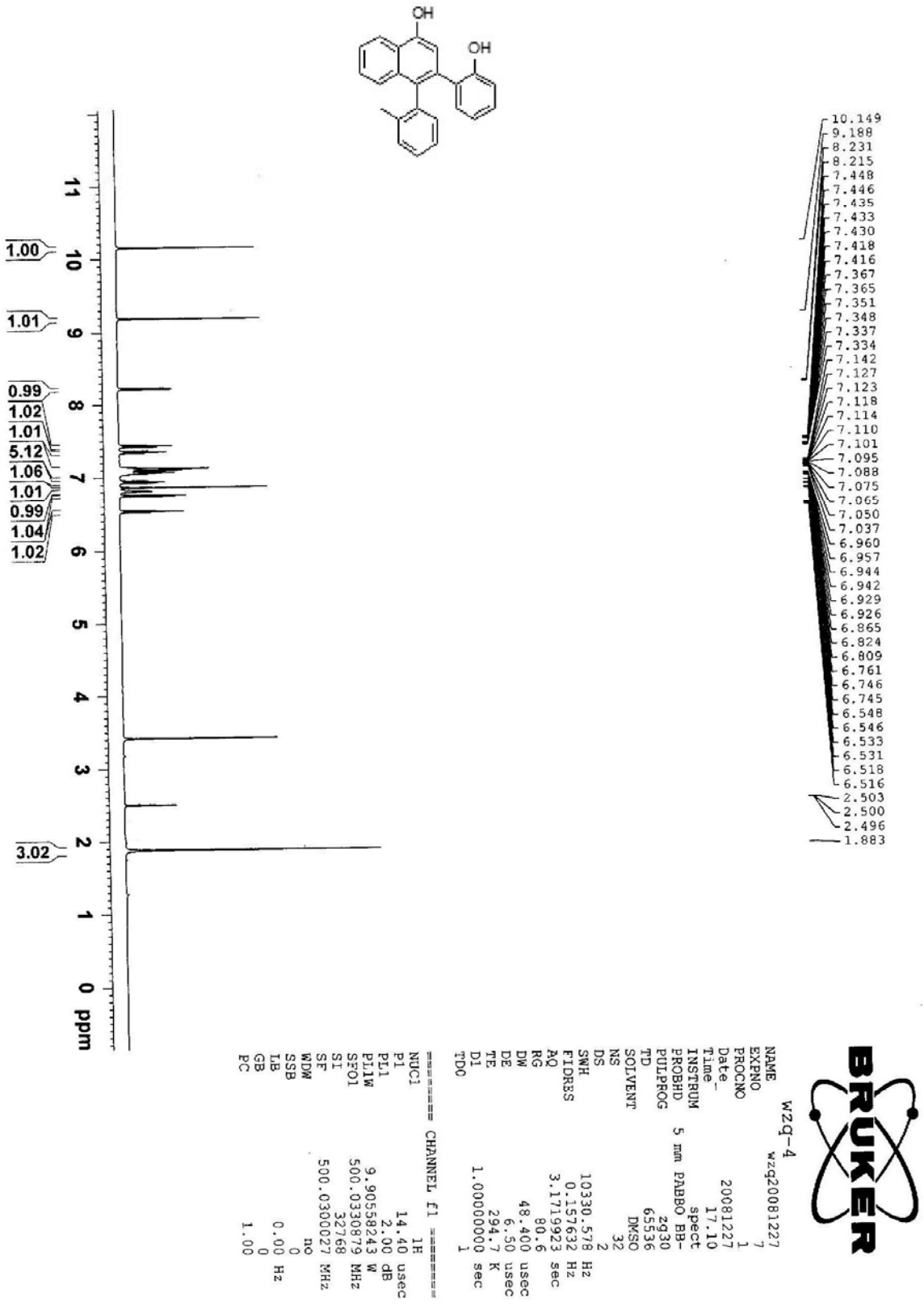
3-(2-Hydroxyphenyl)-4-*m*-tolylnaphthalen-1-ol (2b)



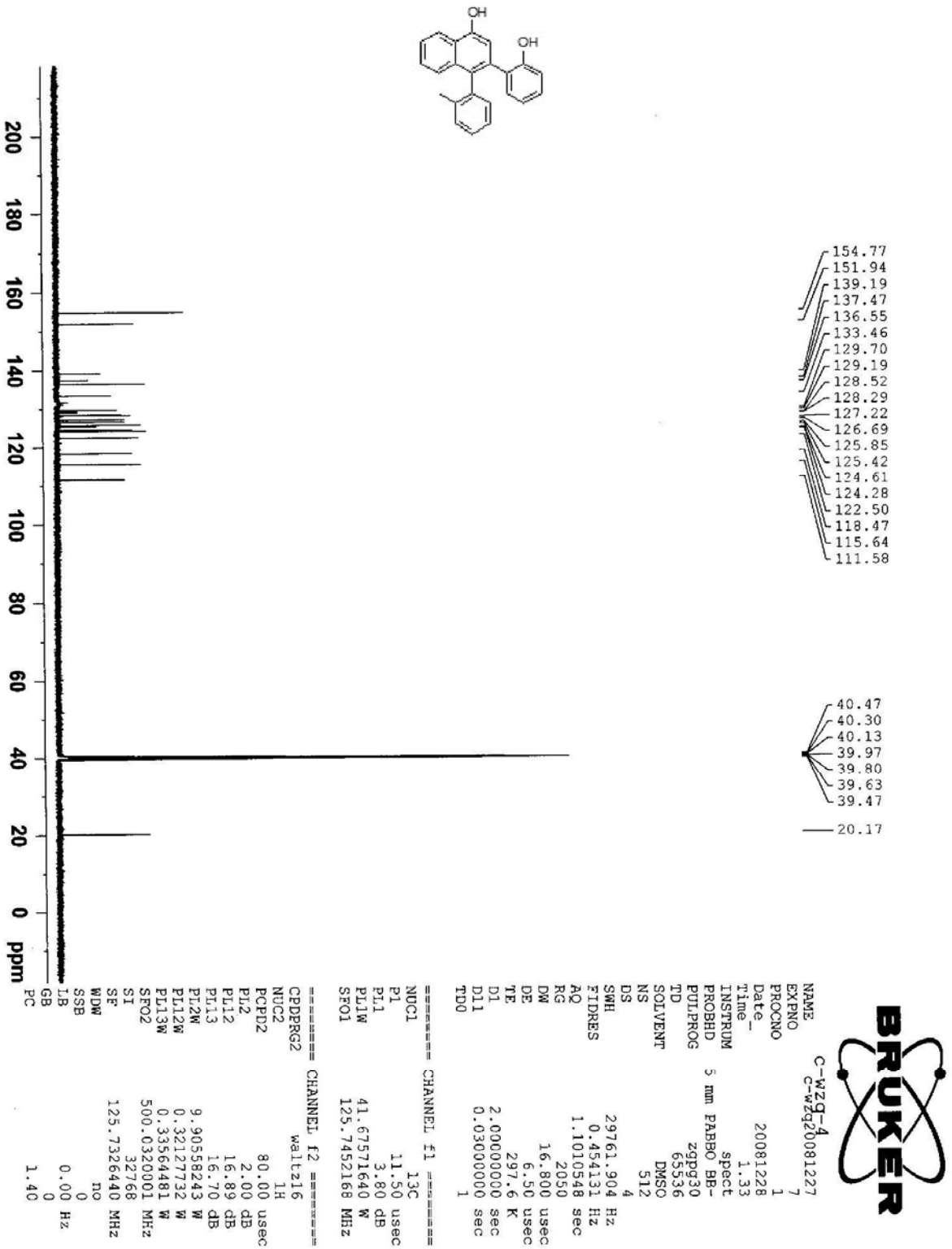
3-(2-Hydroxyphenyl)-4-*m*-tolylnaphthalen-1-ol (2b)



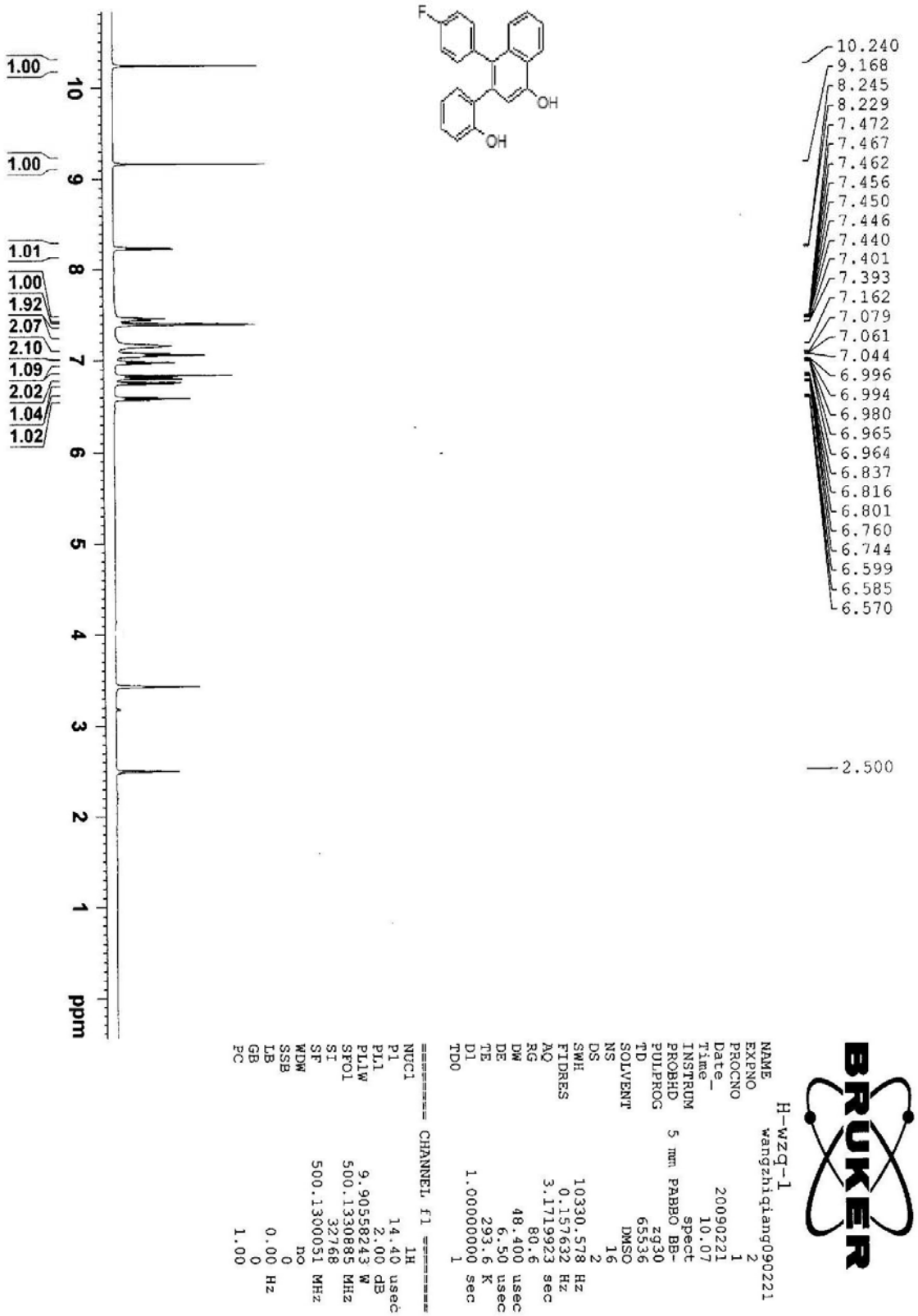
3-(2-Hydroxyphenyl)-4-*o*-tolylnaphthalen-1-ol (2c)



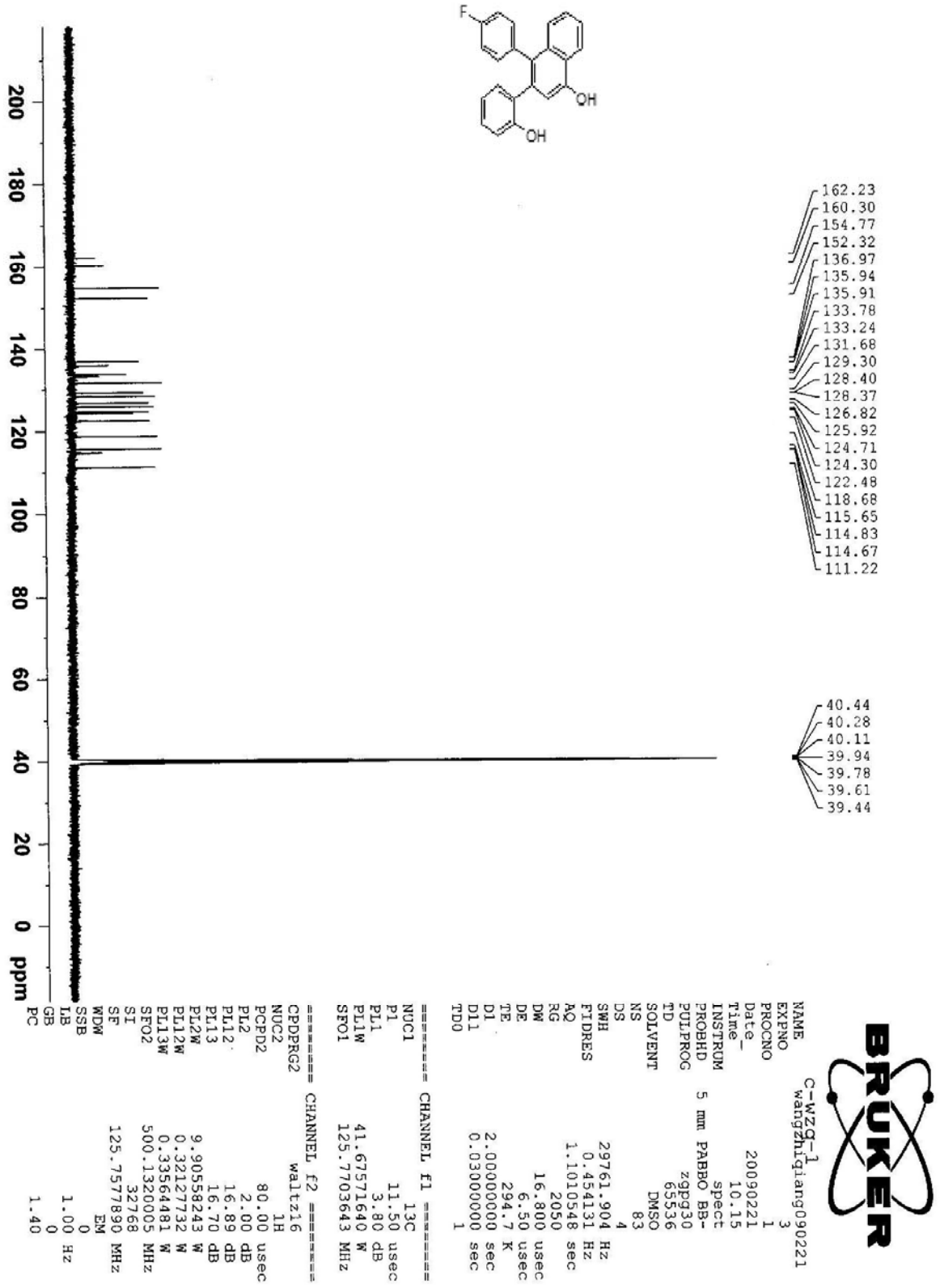
3-(2-Hydroxyphenyl)-4-*o*-tolylnaphthalen-1-ol (2c)



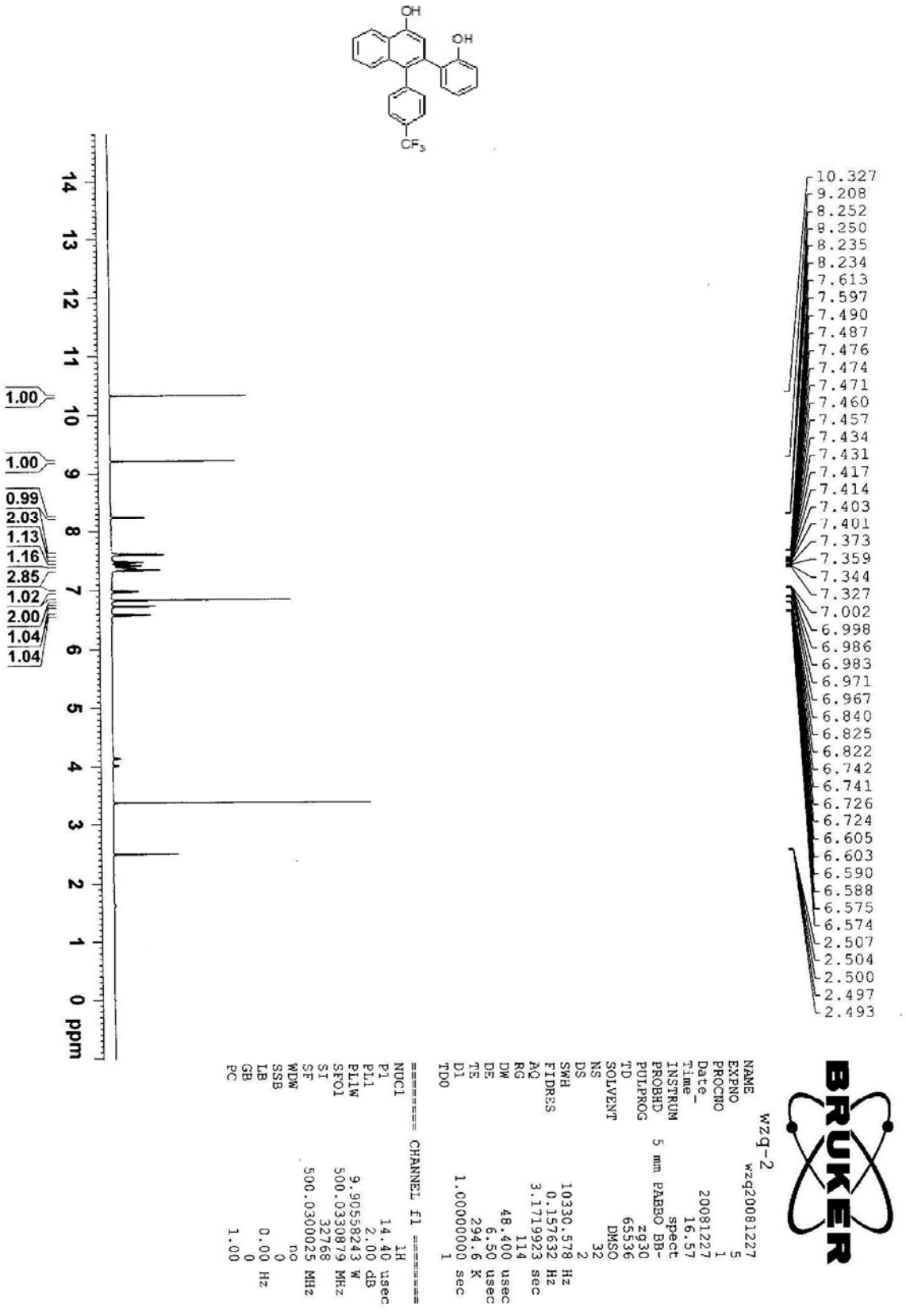
4-(4-Fluorophenyl)-3-(2-hydroxyphenyl)naphthalen-1-ol (2d)



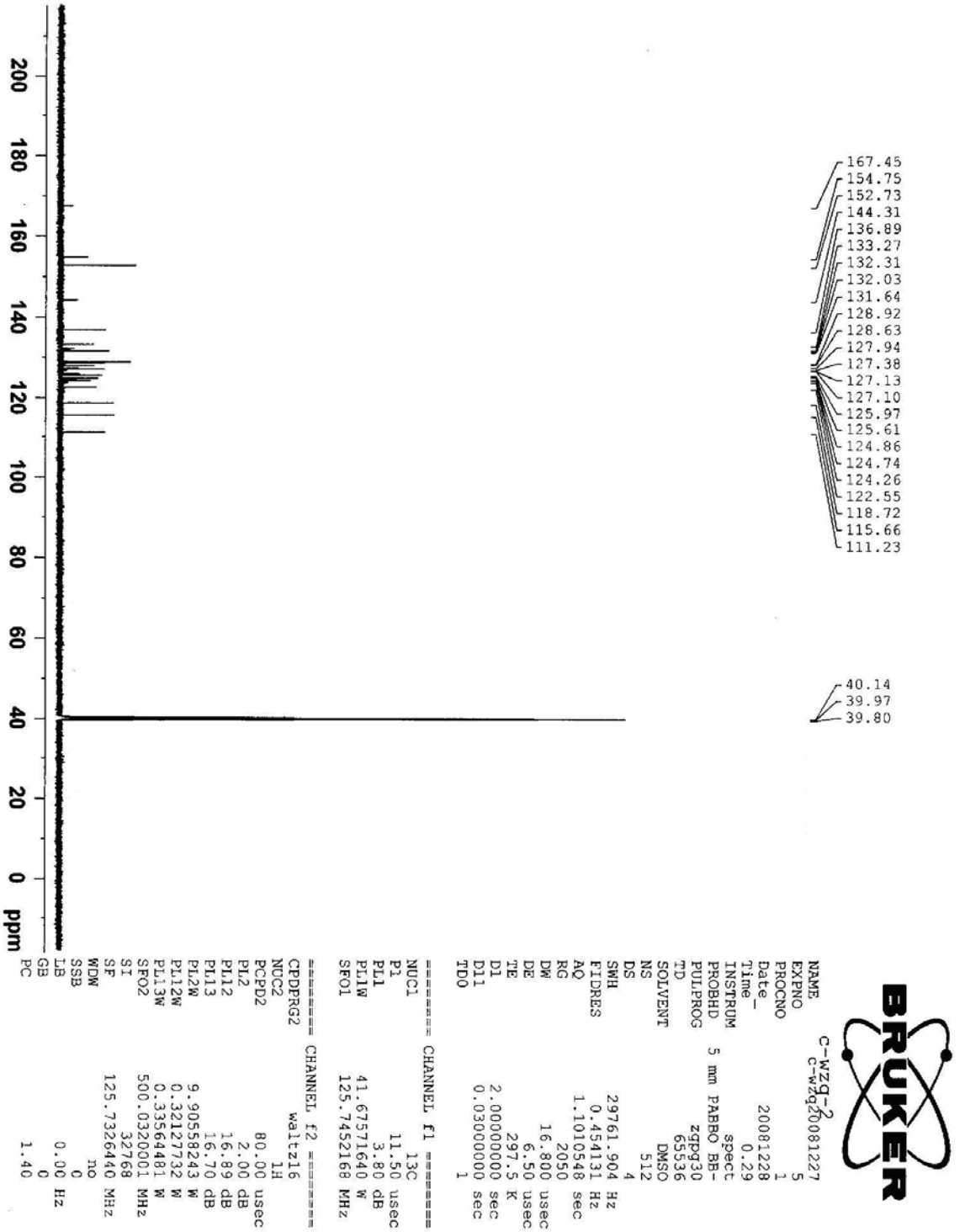
4-(4-Fluorophenyl)-3-(2-hydroxyphenyl)naphthalen-1-ol (2d)



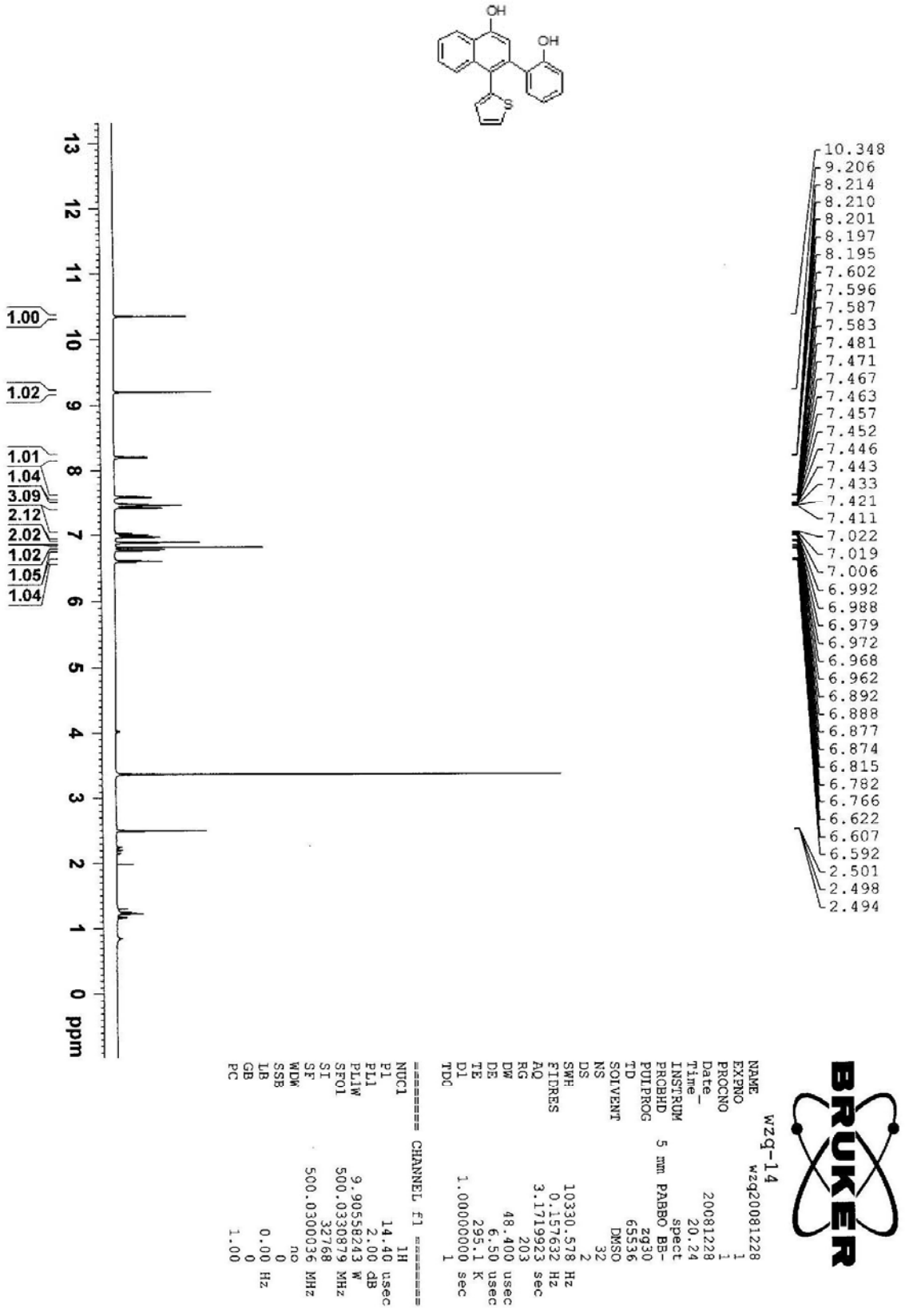
4-(4-(Trifluoromethyl)phenyl)-3-(2-hydroxyphenyl)naphthalen-1-ol (2e)



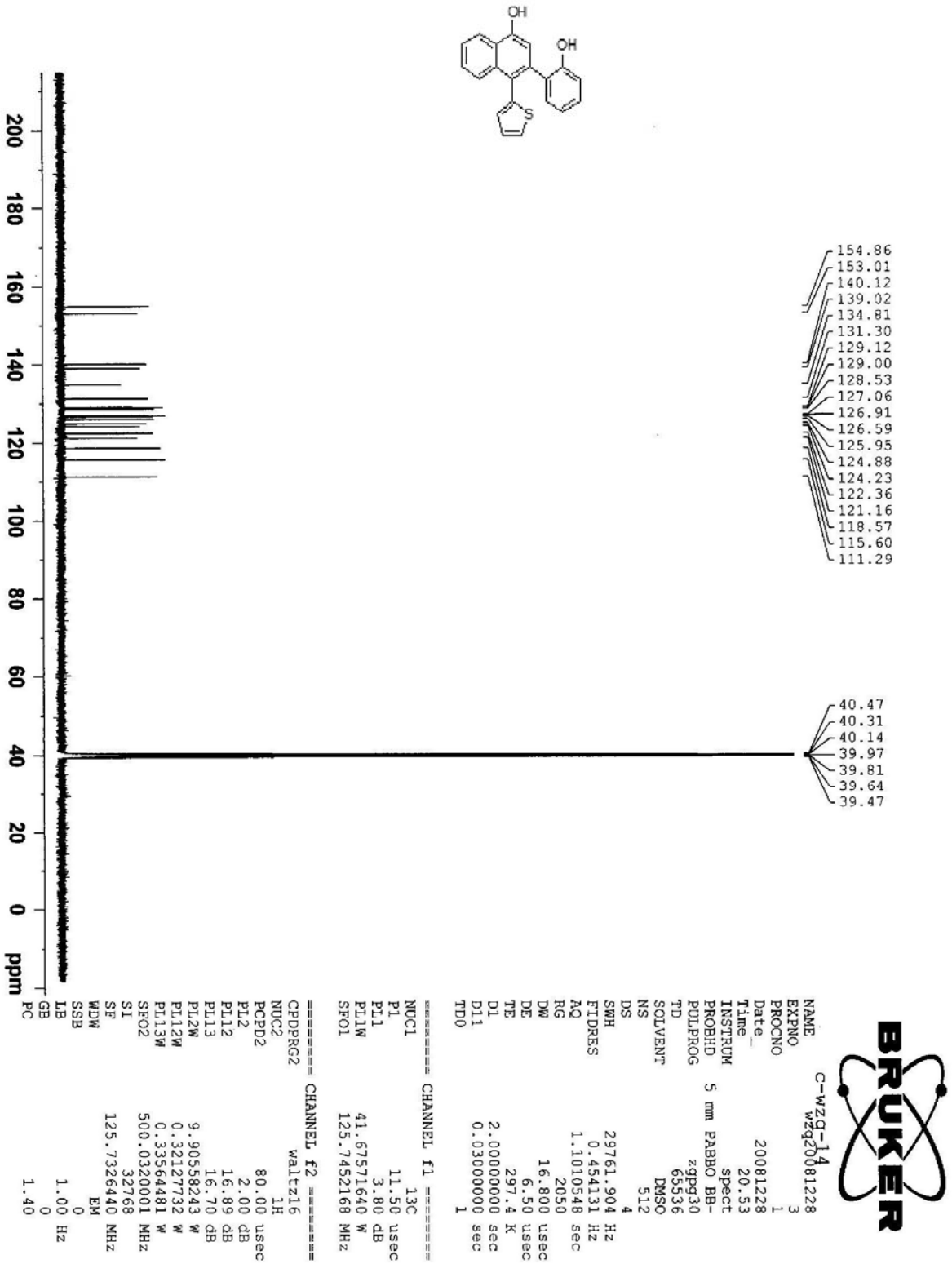
4-(4-(Trifluoromethyl)phenyl)-3-(2-hydroxyphenyl)naphthalen-1-ol (2e)



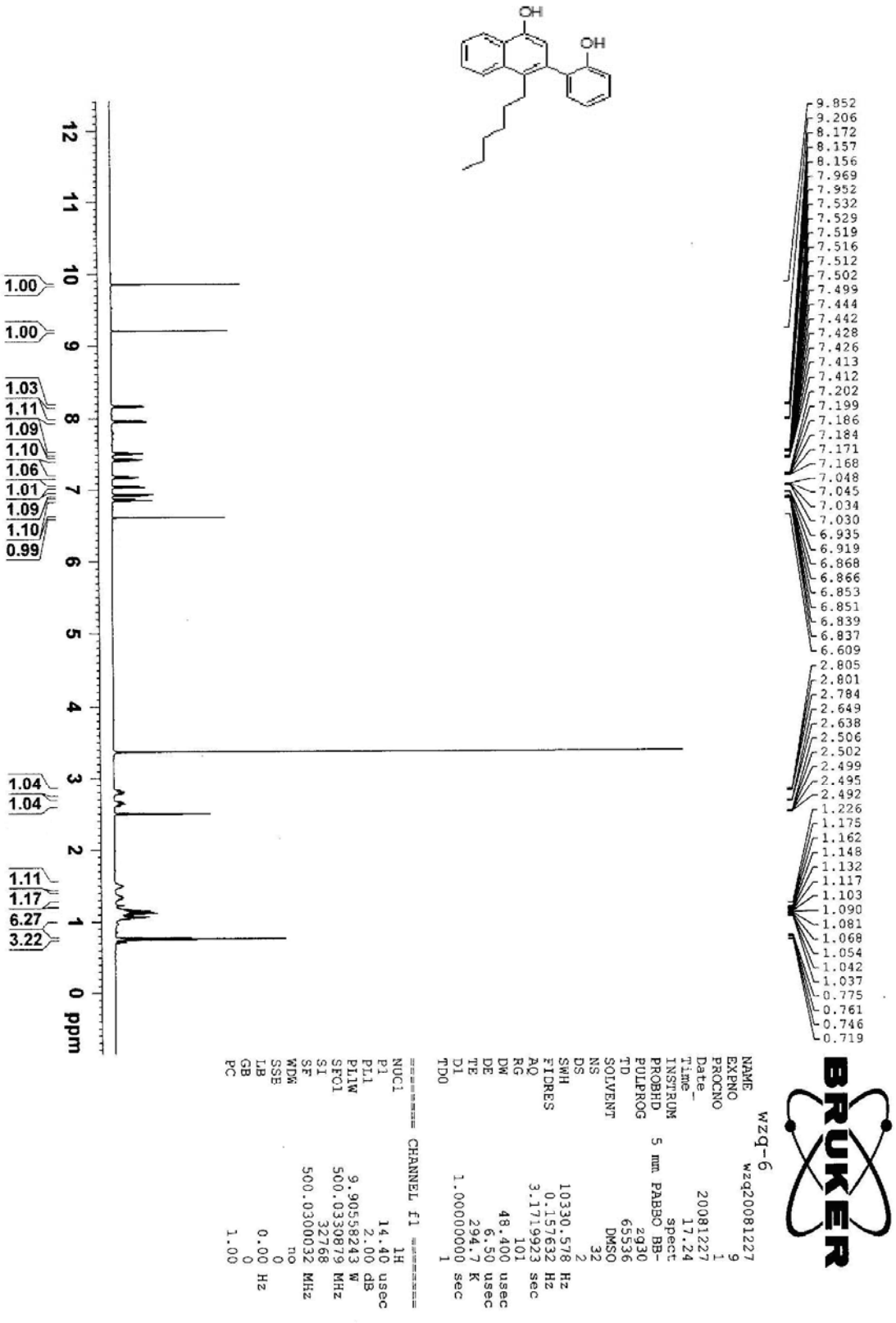
3-(2-Hydroxyphenyl)-4-(thiophen-2-yl)naphthalen-1-ol (2f)



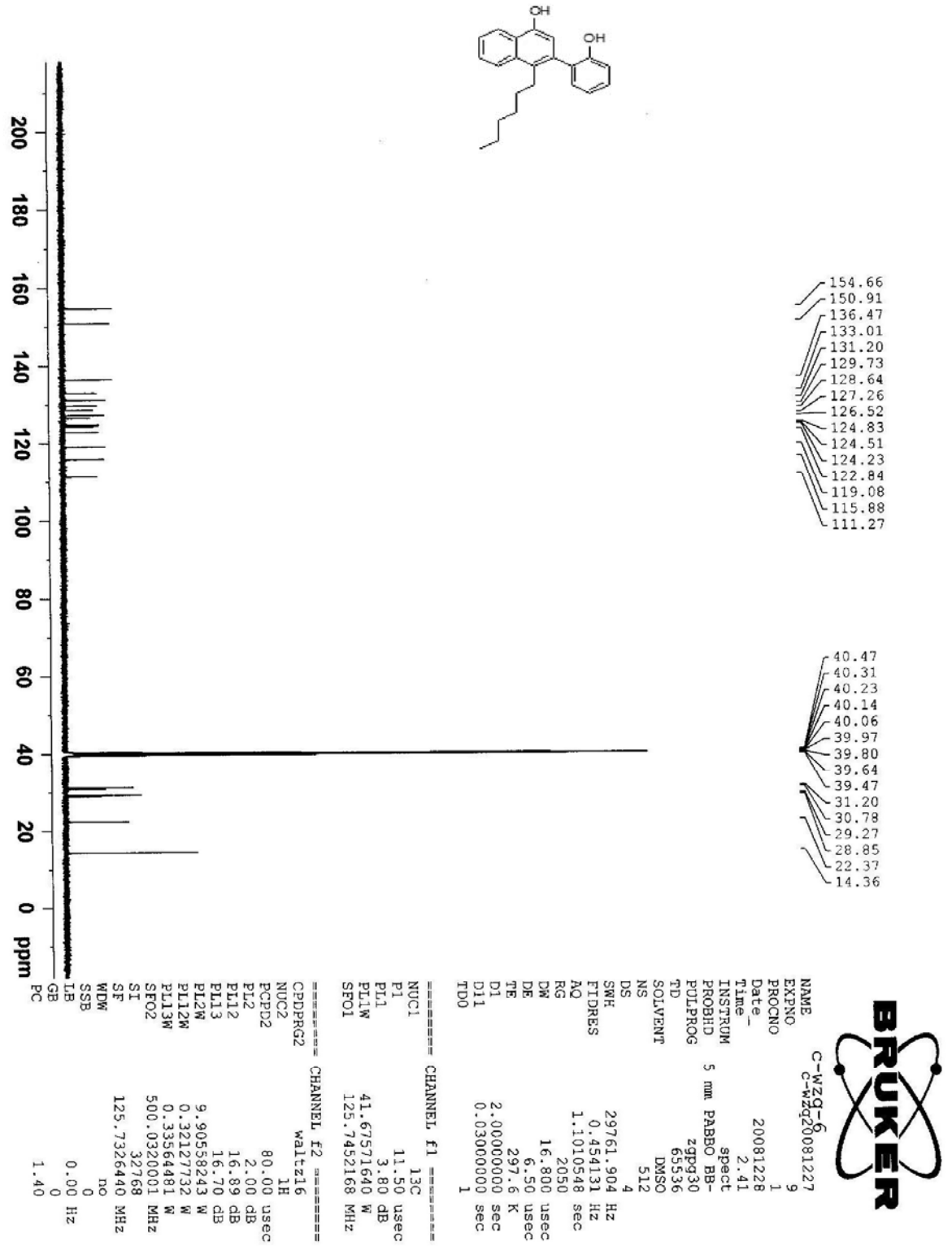
3-(2-Hydroxyphenyl)-4-(thiophen-2-yl)naphthalen-1-ol (2f)



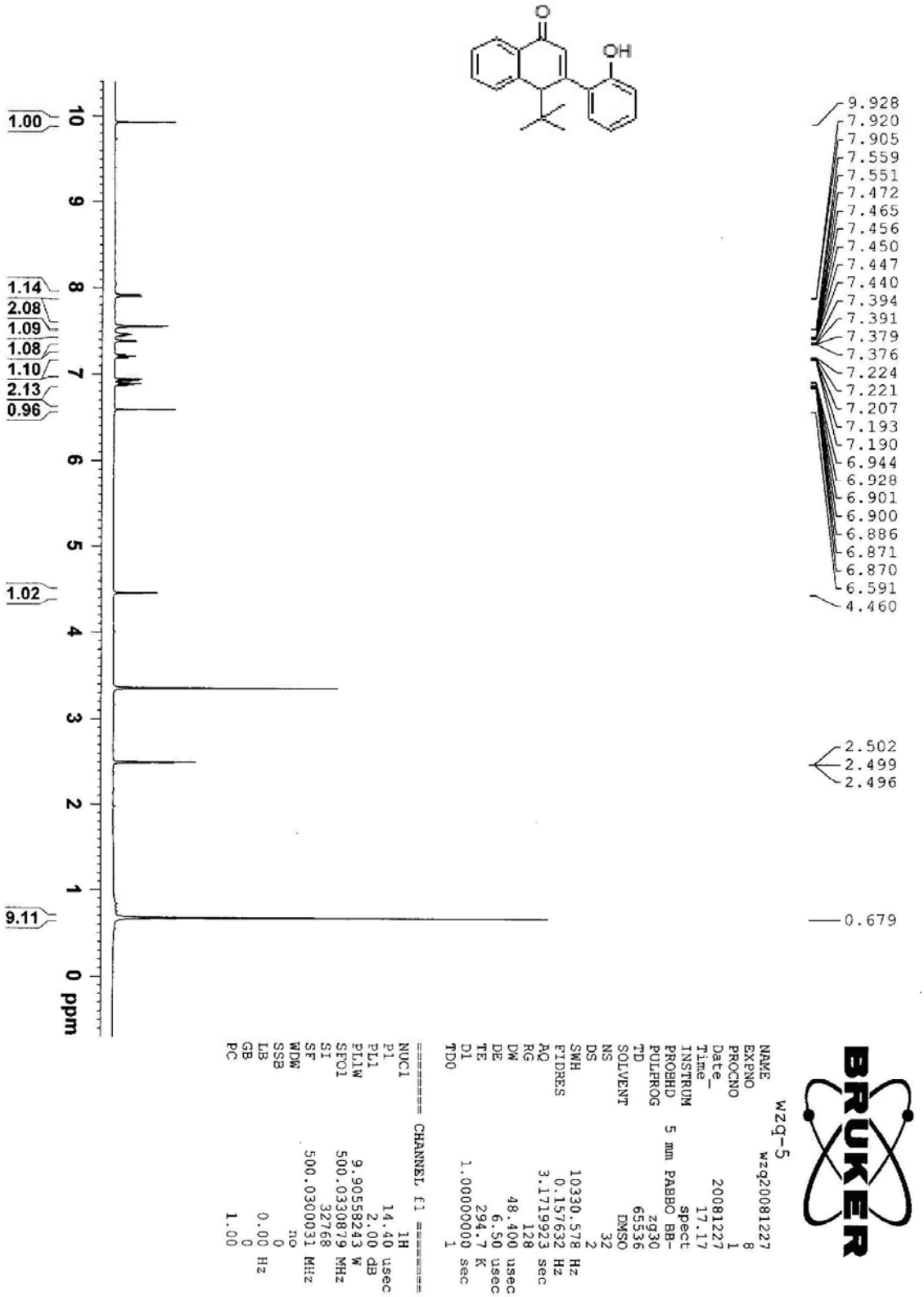
4-Hexyl-3-(2-hydroxyphenyl)naphthalen-1-ol (2g)



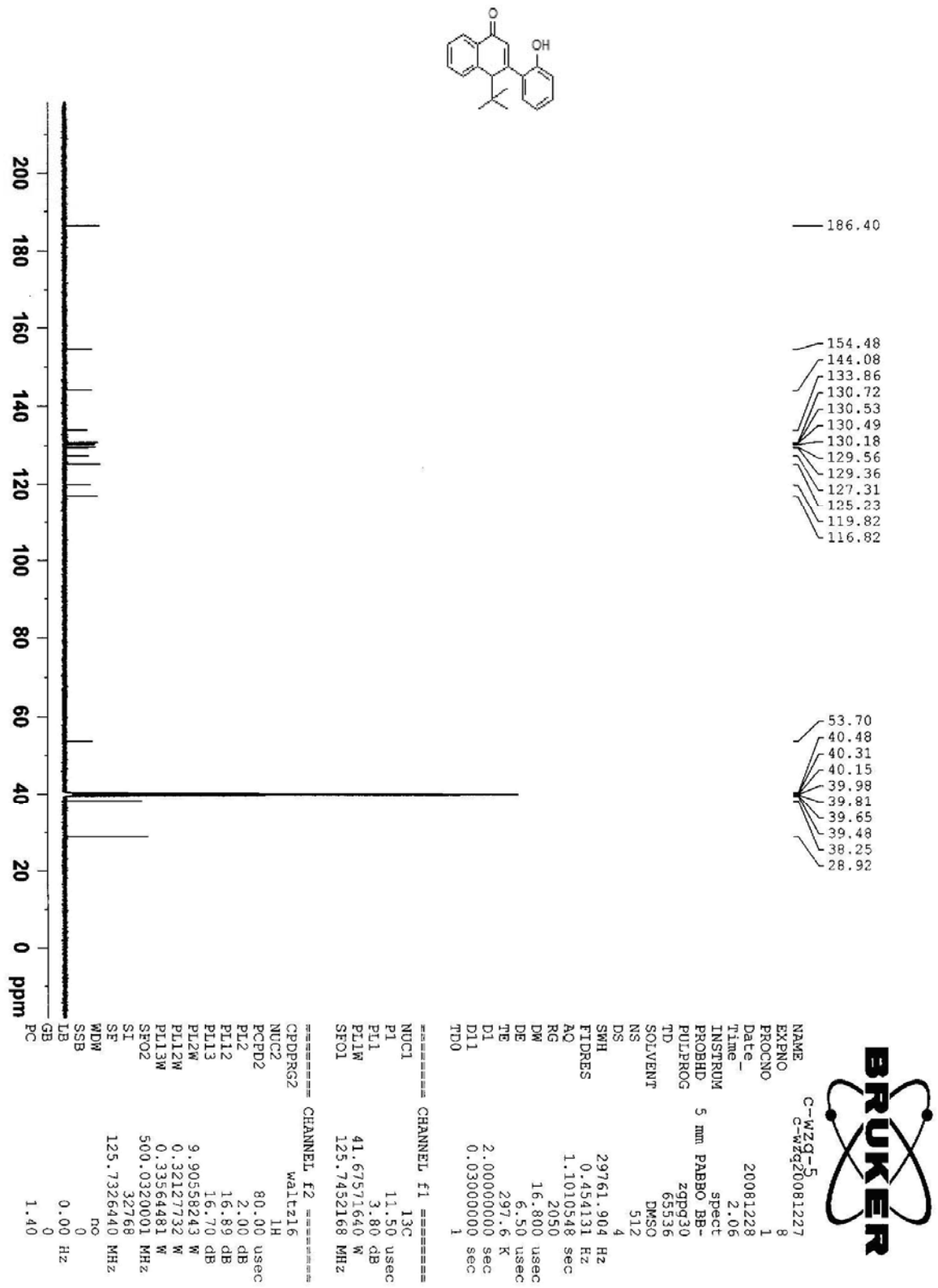
4-Hexyl-3-(2-hydroxyphenyl)naphthalen-1-ol (2g)



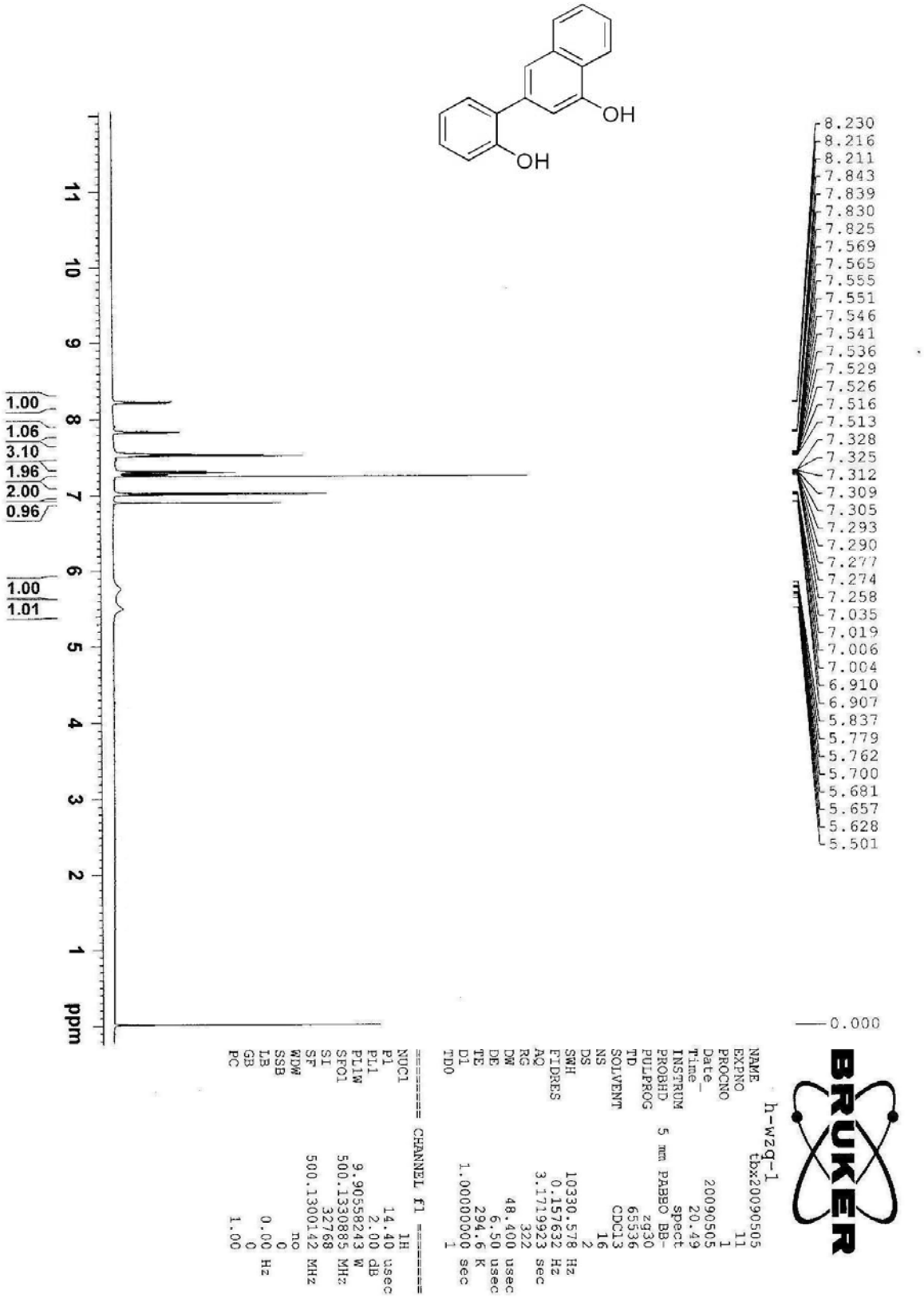
4-*tert*-Butyl-3-(2-hydroxyphenyl)naphthalen-1(4*H*)-one (2h)



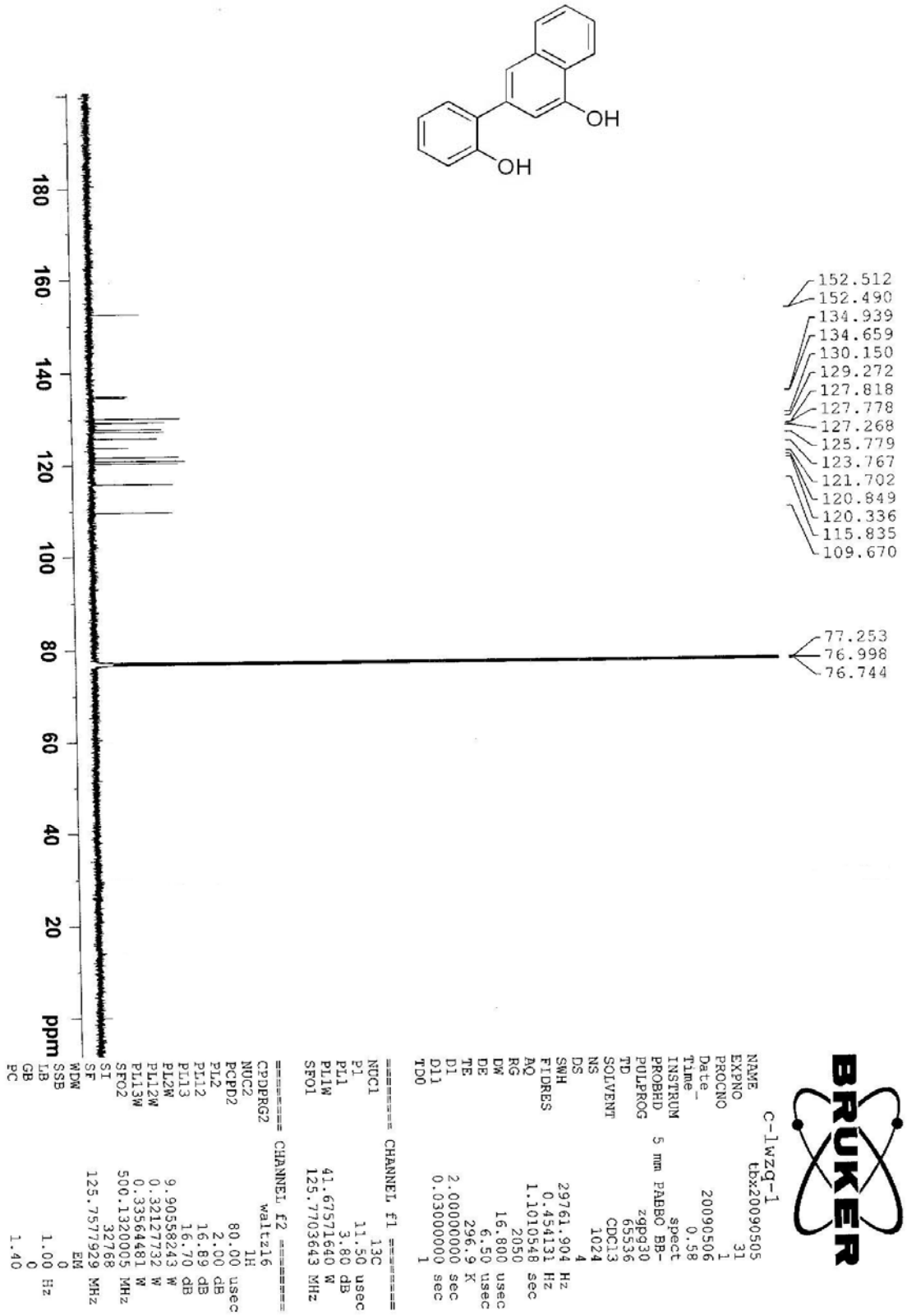
4-*tert*-Butyl-3-(2-hydroxyphenyl)naphthalen-1(4*H*)-one (2h)



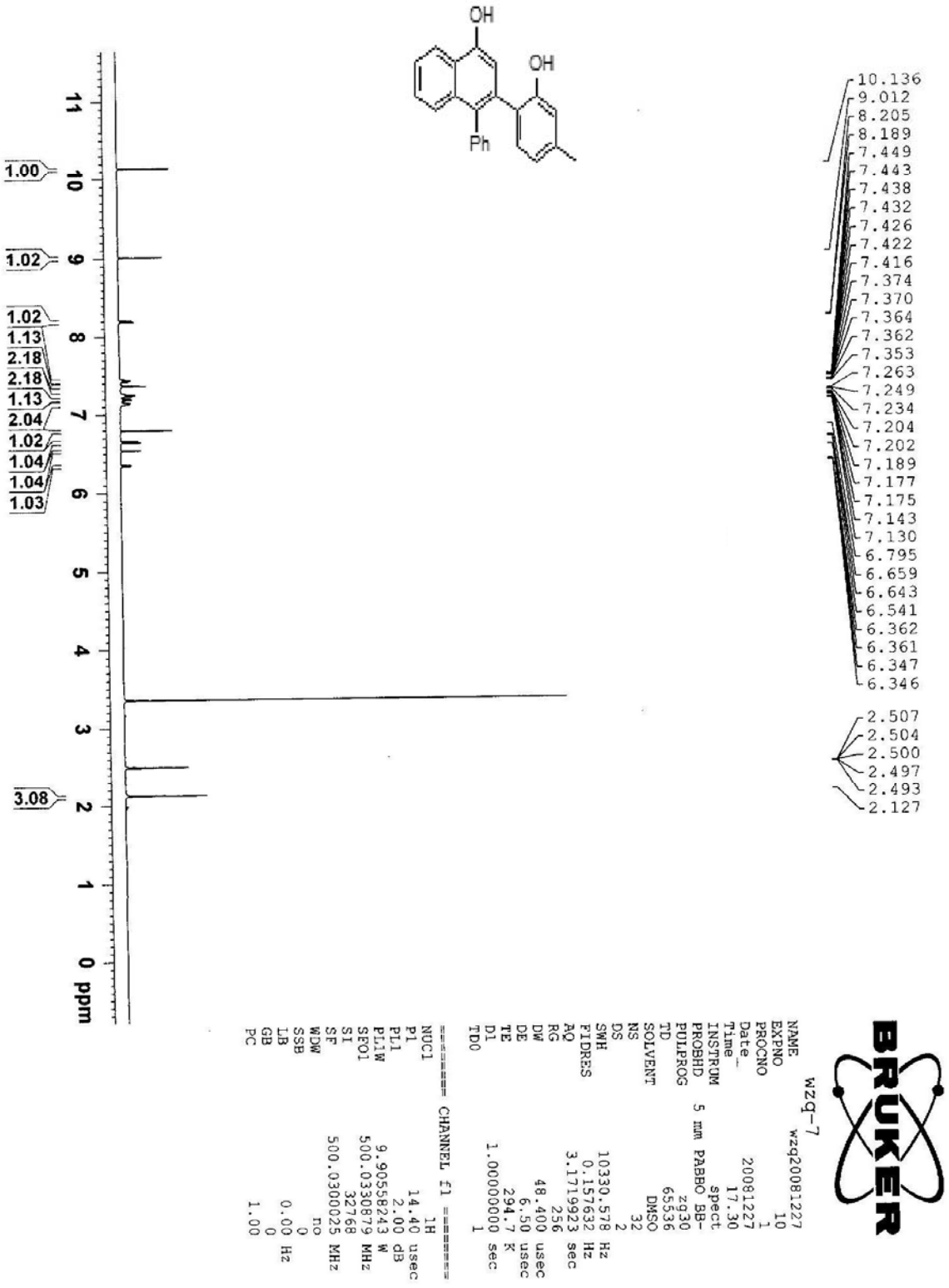
3-(2-Hydroxyphenyl)naphthalen-1-ol (2i)



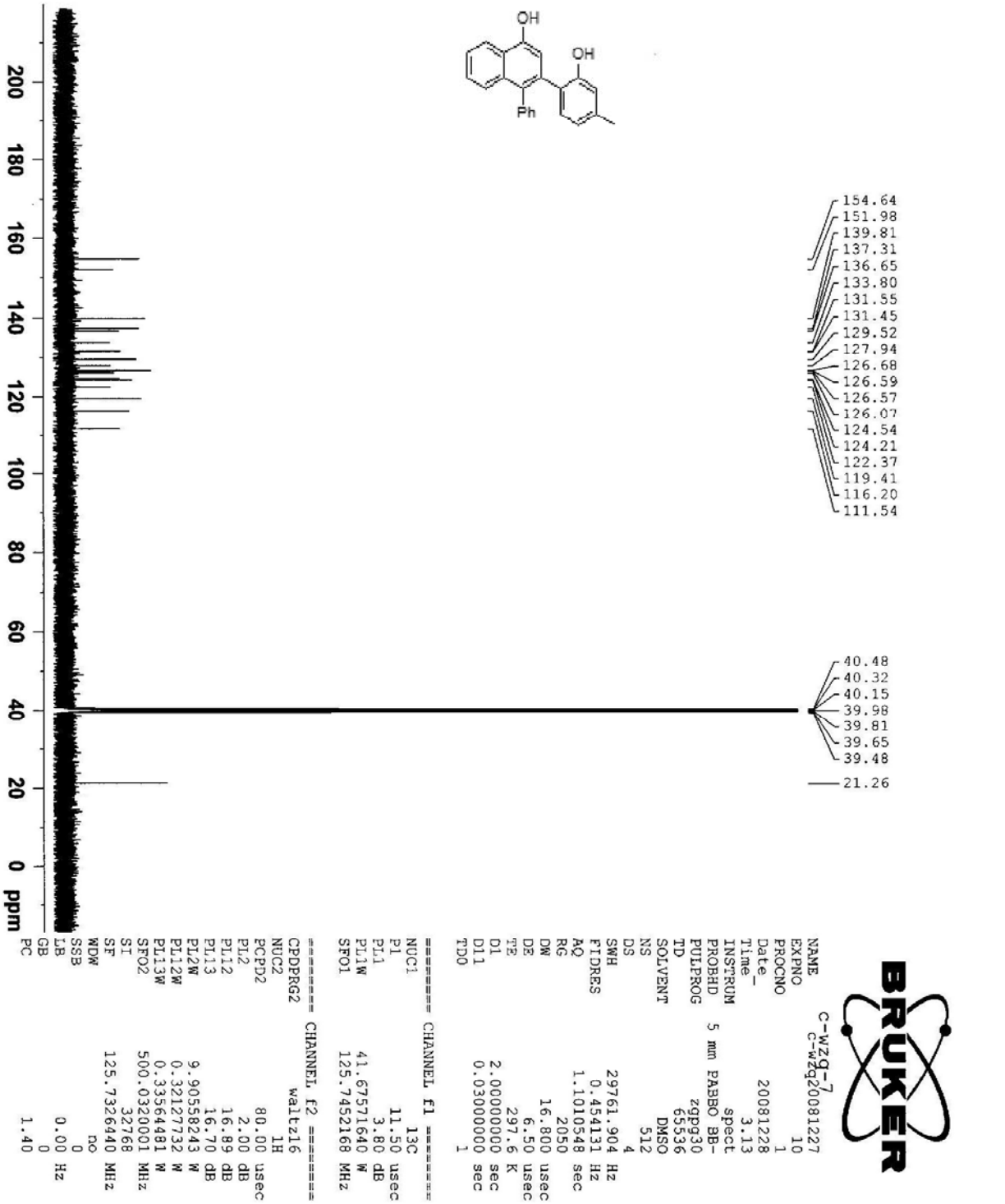
3-(2-Hydroxyphenyl)naphthalen-1-ol (2i)



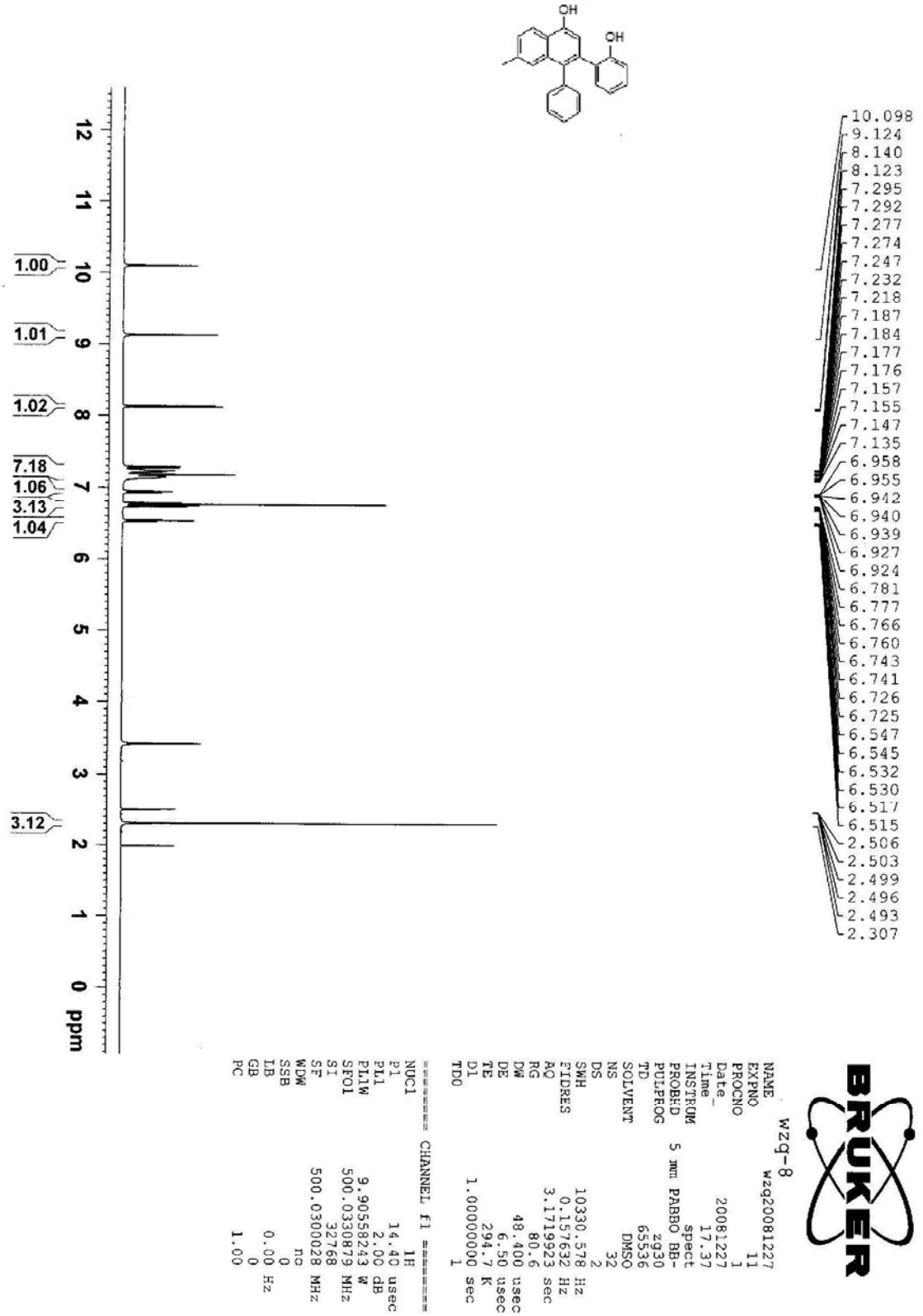
3-(2-Hydroxy-4-methylphenyl)-4-phenylnaphthalen-1-ol (2j)



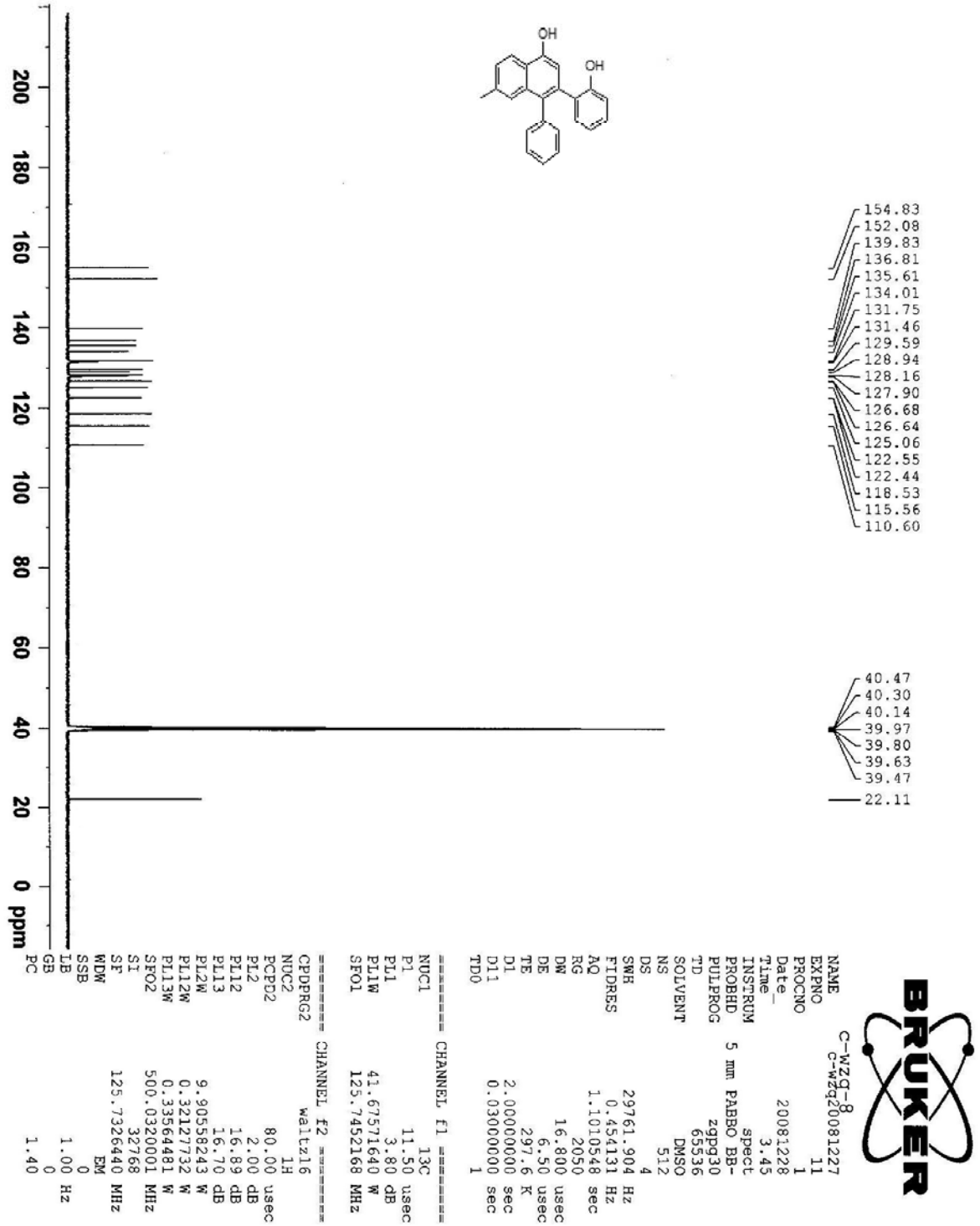
3-(2-Hydroxy-4-methylphenyl)-4-phenylnaphthalen-1-ol (2j)



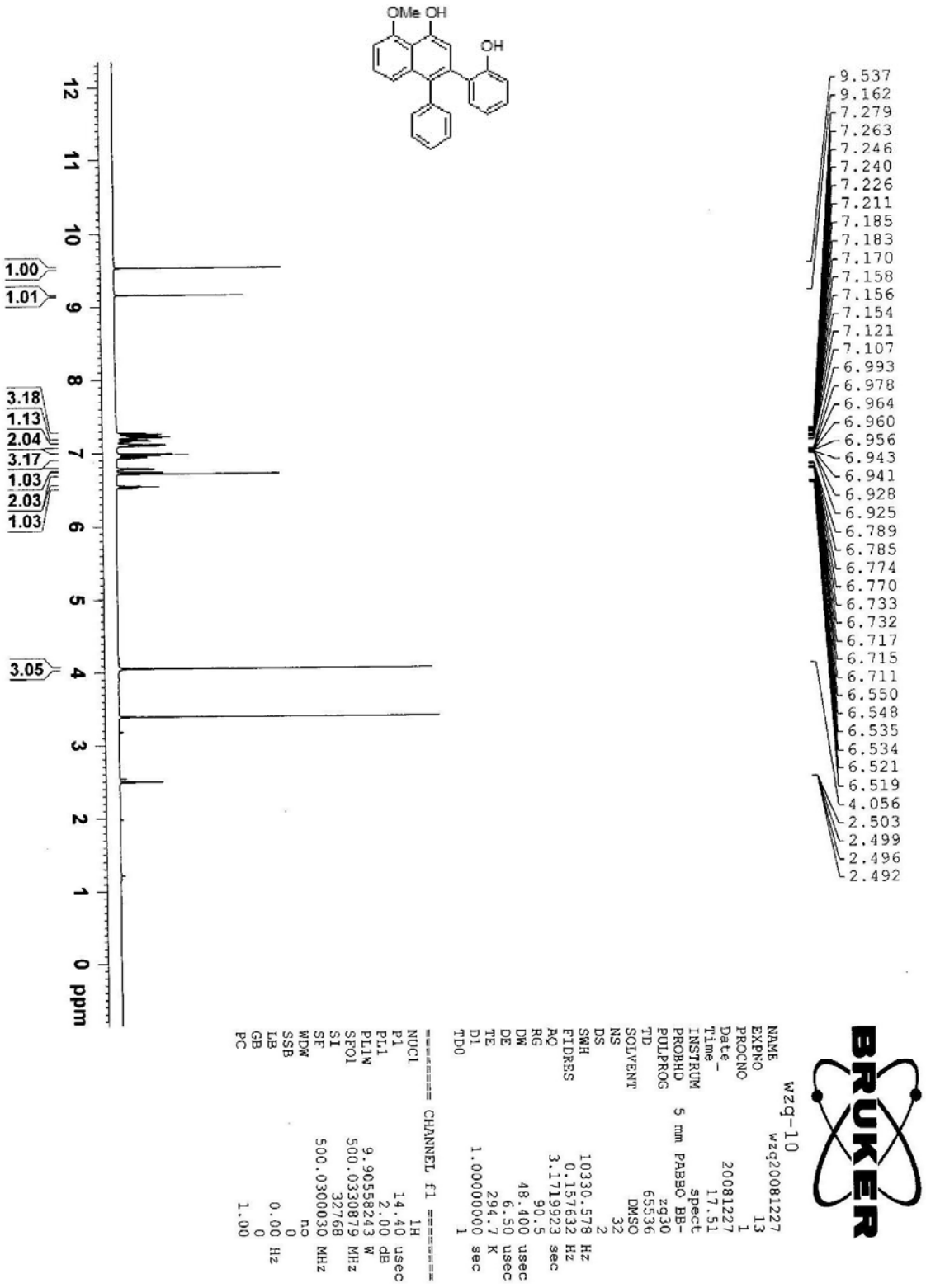
3-(2-Hydroxyphenyl)-6-methyl-4-phenylnaphthalen-1-ol (2k)



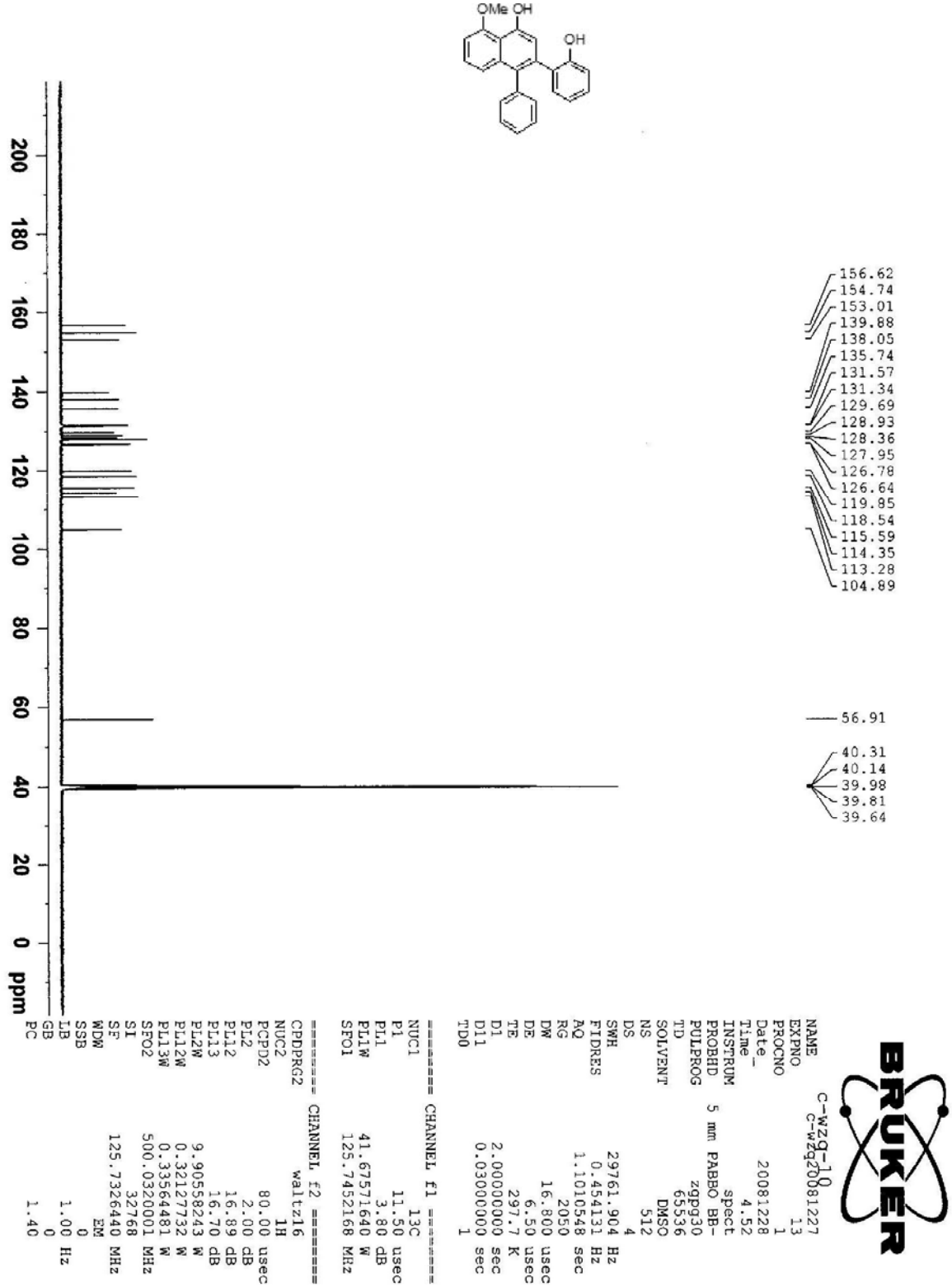
3-(2-Hydroxyphenyl)-6-methyl-4-phenylnaphthalen-1-ol (2k)



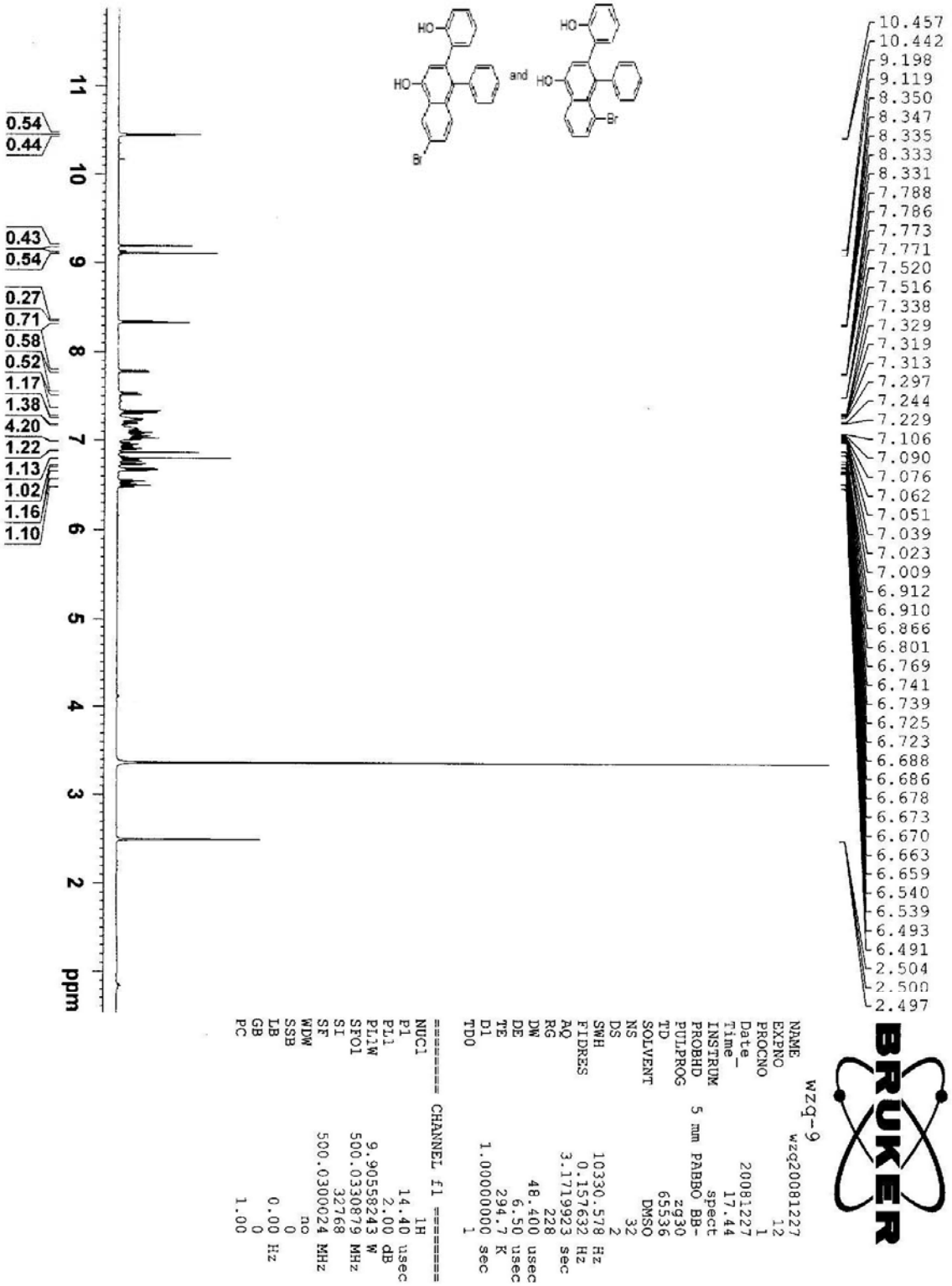
3-(2-Hydroxyphenyl)-8-methoxy-4-phenylnaphthalen-1-ol (2l)



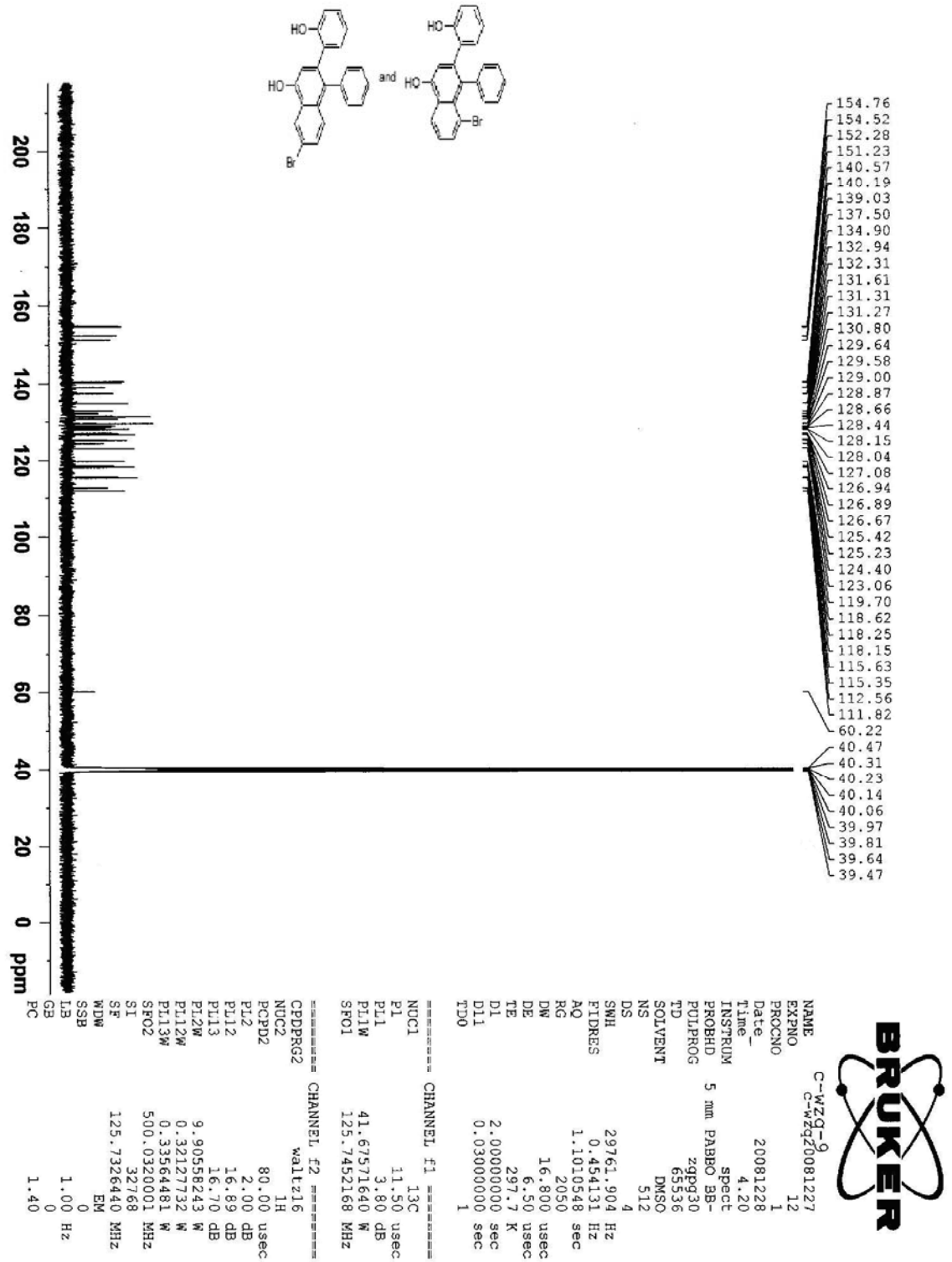
3-(2-Hydroxyphenyl)-8-methoxy-4-phenylnaphthalen-1-ol (2l)



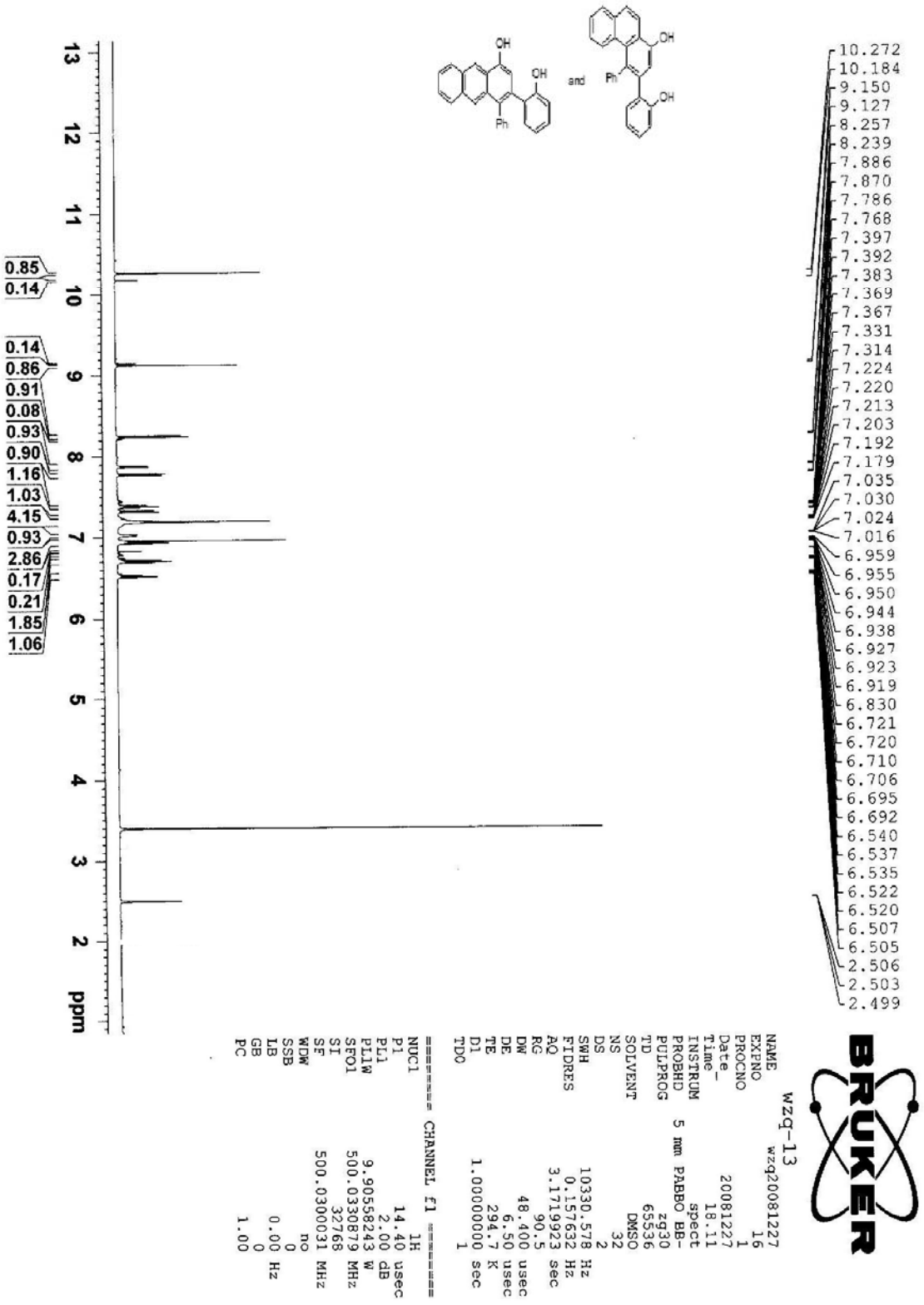
7-Bromo-3-(2-hydroxyphenyl)-4-phenylnaphthalen-1-ol (2m) and 5-bromo-3-(2-hydroxyphenyl)-4-phenylnaphthalen-1-ol (2m′)



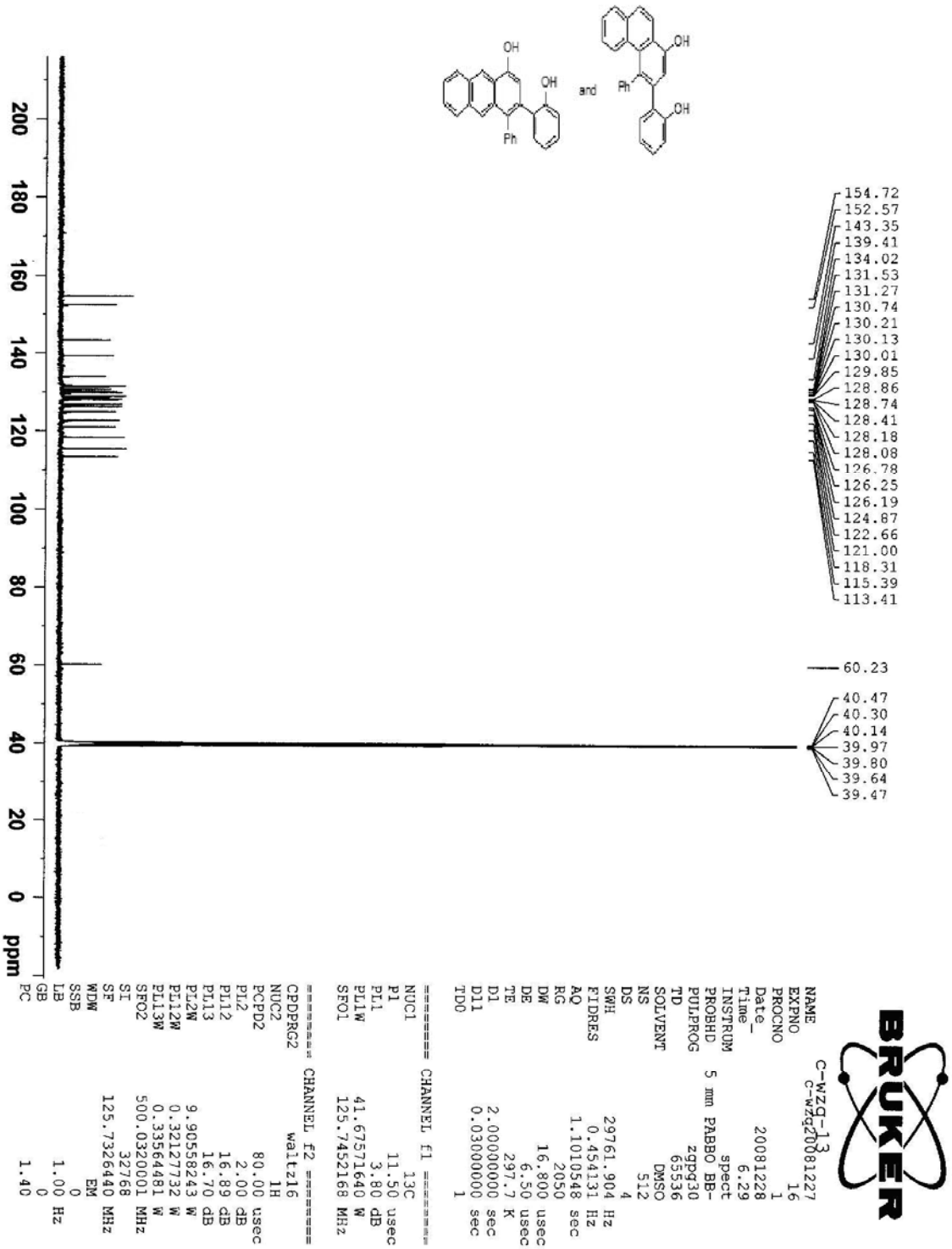
7-Bromo-3-(2-hydroxyphenyl)-4-phenylnaphthalen-1-ol (2m) and 5-bromo-3-(2-hydroxyphenyl)-4-phenylnaphthalen-1-ol (2m′)



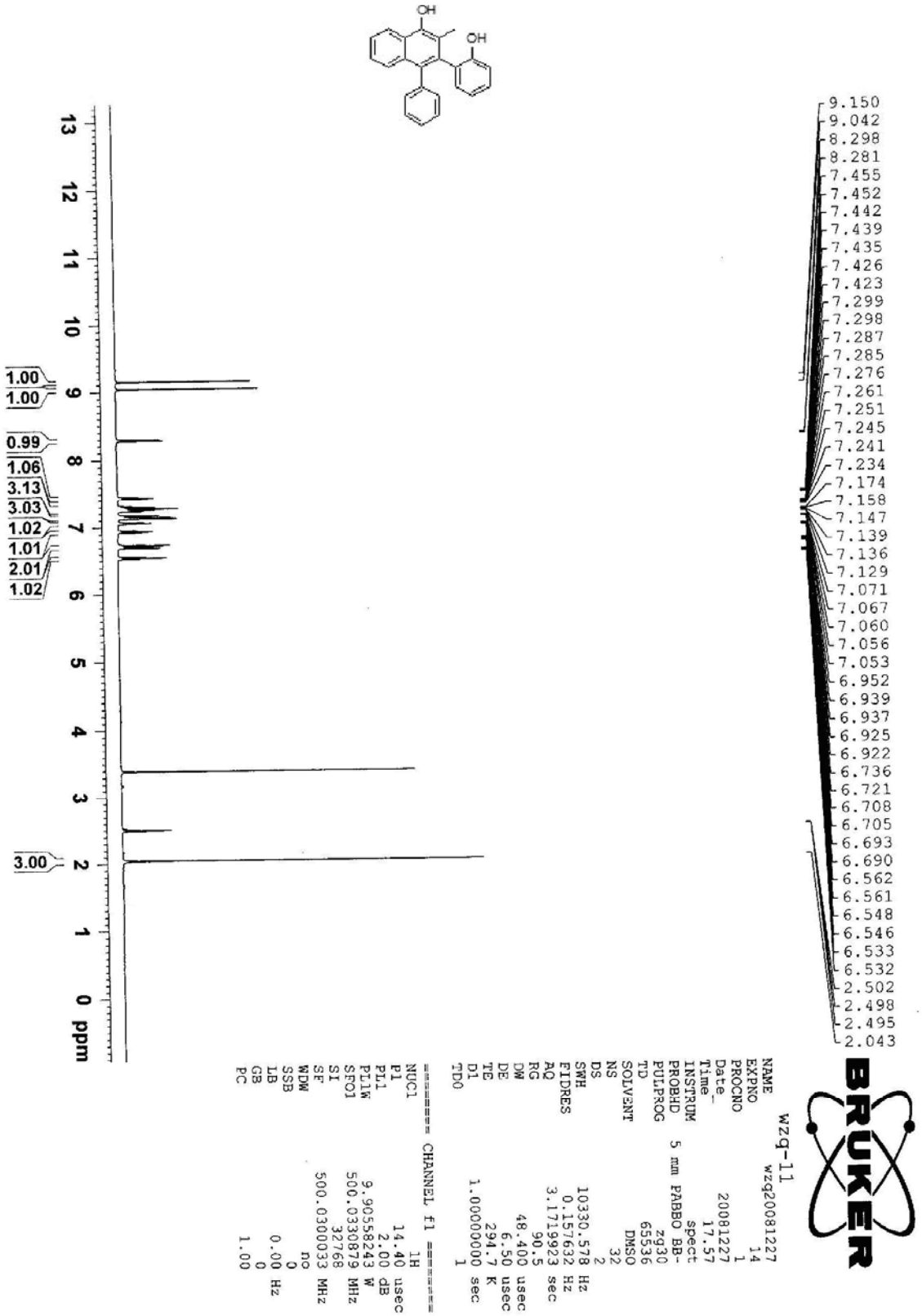
3-(2-Hydroxyphenyl)-4-phenylanthracen-1-ol (2n) and 3-(2-hydroxyphenyl)-4-phenylphenanthren-1-ol (2n′)



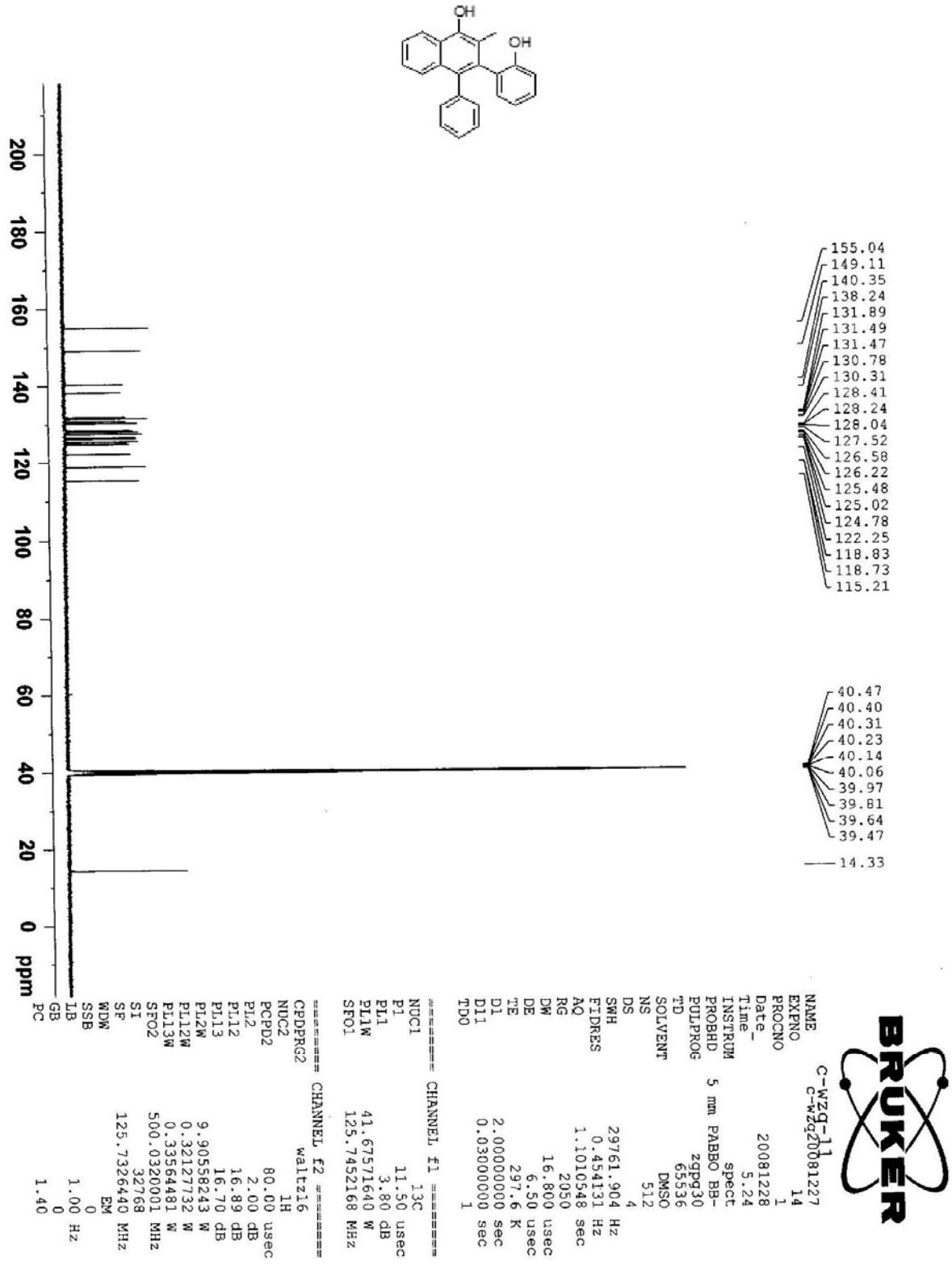
3-(2-Hydroxyphenyl)-4-phenylanthracen-1-ol (2n) and 3-(2-hydroxyphenyl)-4-phenylphenanthren-1-ol (2n′)



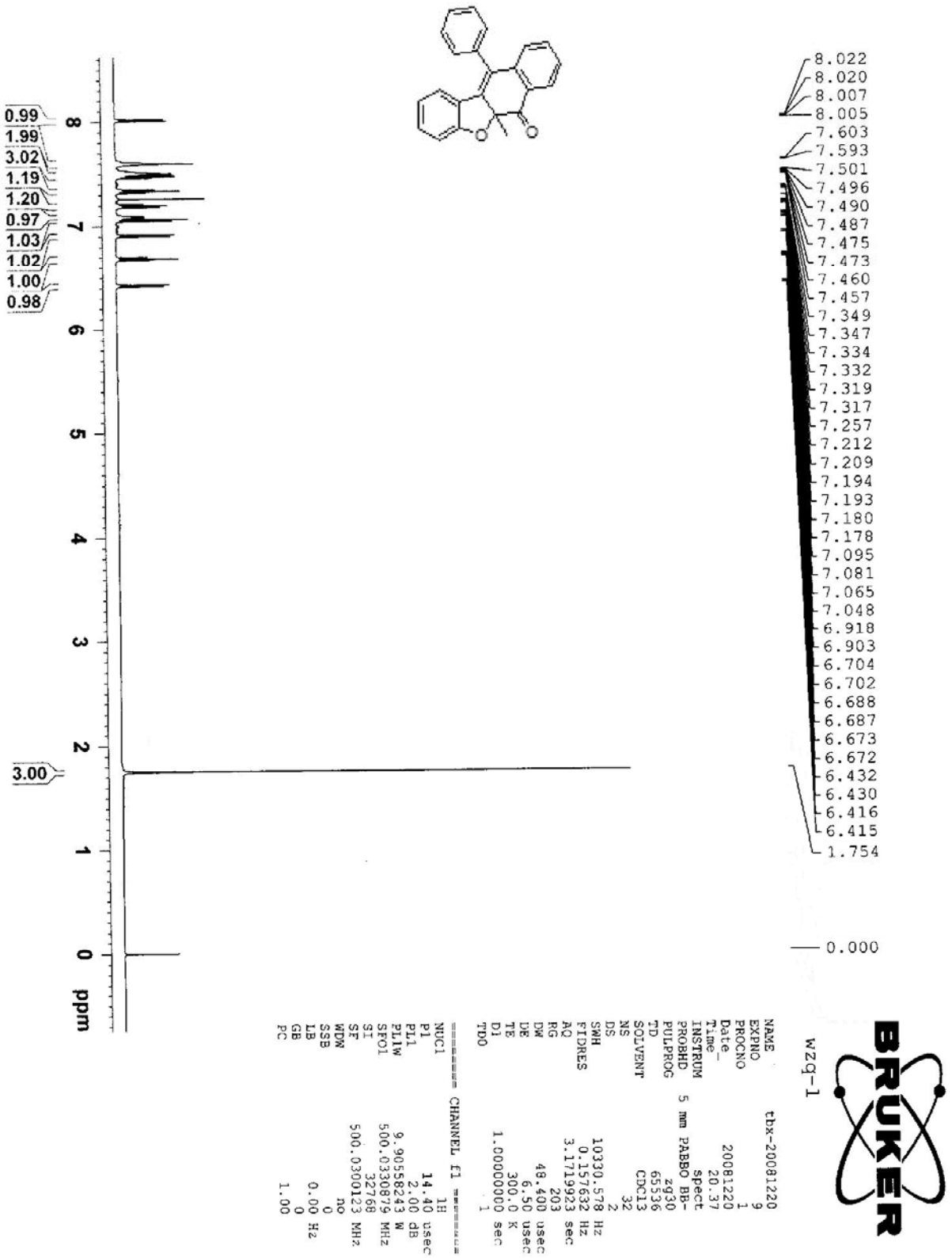
3-(2-Hydroxyphenyl)-2-methyl-4-phenylnaphthalen-1-ol (2o)



3-(2-Hydroxyphenyl)-2-methyl-4-phenylnaphthalen-1-ol (2o)



The product (3o)



The product (3o)