Electronic Supplementary Material (ESI) for RSC Advances.

Supporting Information

Calcium Catalysed Regioselective Tandem Process for the Synthesis of Fully Substituted Furans

Srinivasarao Yaragorla,* Ravikrishna Dada, Abhishek Pareek and Garima Singh

SY-Organic & Medicinal Chemistry Laboratory, Department of Chemistry, Central University of Rajasthan, NH-8, Bandersindri, Ajmer distt, 305817, Rajasthan, India. E-mail: srinivasarao@curaj.ac.in or ysriict@gmail.com

List of contents

1. General information

The starting materials were synthesized and used. ^{1}H, ^{13}C NMR spectra were recorded on avance bruker 500 MHz spectrometer, in $CDCl_3$. Chemical shifts (δ) are given in ppm relative to tetramethylsilane (TMS) and calibrated to residual chloroform peaks. Coupling constants (*J*) are reported in Hz and coupling patterns are described as s = singlet, d = doublet, t = triplet, q = quartet, quint= quintet, hept = heptet, m = multiplet. Mass spectra were recorded on a Agilent 6530 Accurate- Mass Q-TOF [electron ionization (EI), 70 eV] and peaks are listed according to their m/z values. Melting points were measured with a Büchi Melting Point B-540 apparatus. Column chromatography was performed with Merck silica gel 60 (60-120 mesh). Reactions were monitored by thin layer chromatography (TLC) with aluminium sheets silica gel 60 F254 from Merck with detection by UV light and charring with $KMnO_4$ stain.

2. General procedure for the syntheses of propargylic alcohol (1)[1]

LHMDS (1.2 equiv.) was added to a stirred solution of alkyl propiolate or phenyl acetylene (1.1 equiv.) in dry THF (15 ml.) at -78 ºC and stirred for 30 minutes at the same temperature. Aryl aldehyde (1 equiv.) was added slowly to the reaction mixture and the reaction progress was monitored by TLC. After completion, the reaction mixture was quenched with saturated NH_4Cl and extracted into EtOAc thrice. The combined organic layers were washed with brine, dried over anhydrous Na_2SO_4, solvent was removed under reduced pressure and the crude product was purified by silica gel column chromatography (pet ether: EtOAc) to obtain the desired product (1).

3. General procedure for the syntheses of fully substituted furocoumarins (3a-3i, 6a-6c and 7a-7c):

A mixture of propargylic alcohol (**1**, 1.0 equiv.,) and 4-hydroxy-coumarin (**2**, 1.1 equiv.) were heated at 120 ^{o}C in presence of $Ca(OTf)_2$ (10 mol%) and Bu_4NPF_6 (10 mol%). Heating was continued till completion of the reaction (monitored by TLC, generally 2-3.5 h). After completion reaction mixture was brought to room temperature and was diluted with minimum amount water and

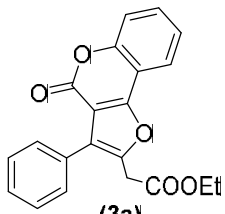
extracted with EtOAc thrice. The combined organic layers were washed with brine, dried over anhydrous Na_2SO_4. Solvent was removed under reduced pressure and the crude product was purified by silica gel column chromatography (pet ether: EtOAc) to obtain the desired product.

4. General procedure for the synthesis of fully substituted benzofurans (5a-5o and 8a-8d):

A mixture of propargylic alcohol (**1**, 1.0 equiv.,) and cyclohexane 1,3-dione (**4**, 1.1 equiv.) were heated at 120 ^oC in presence of $Ca(OTf)_2$ (10 mol%) and Bu_4NPF_6 (10 mol%). Heating was continued till completion of the reaction (monitored by TLC, generally 2-4 h). After completion reaction mixture was brought to room temperature and was diluted with minimum amount water and extracted with EtOAc thrice. The combined organic layers were washed with brine, dried over anhydrous Na_2SO_4. Solvent was removed under reduced pressure and the crude product was purified by silica gel column chromatography (pet ether: EtOAc) to obtain the desired product.

5. Characterization data for substituted furans

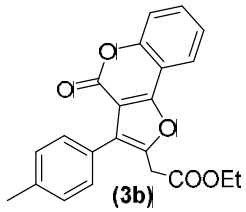
Ethyl 2-(4-oxo-3-phenyl-4*H*-furo[3,2-*c*]chromene-2-yl)acetate (3a)[3]



(3a)

Light yellow solid, m.p. 107 ^oC, 73.7 mg, 91% yield, (eluent: Petroleum ether : EtOAc 88:12) ; ^1H NMR (500 MHz, $CDCl_3$) δ 7.91 (dd, J = 7.5 Hz, J = 8 Hz, 1H), 7.55 - 7.51 (m, 3H), 7.49 - 7.45 (m, 3H), 7.44 - 7.42 (m, 1H), 7.37 - 7.34 (m, 1H), 4.24 (q, J = 7 Hz, 2H), 3.86 (s, 2H), 1.30 (t, J = 7 Hz, 3H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) : δ 168.75, 157.5, 157.2, 152.6, 147.3, 130.8, 129.8, 129.2, 128.8, 128.4, 126.6, 124.4, 121.0, 117.2, 112.6, 109.6, 61.7, 32.9, 14.1 ppm.

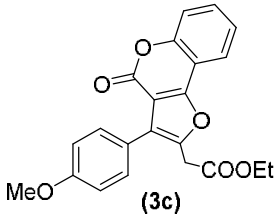
Ethyl 2-(4-oxo-3-(p-tolyl)-4*H*-furo[3,2-*c*]chromene-2-yl)acetate (3b)



(3b)

Brown solid, m.p. 107 °C, 66.4 mg, 80% yield, (eluent: Petroleum ether: EtOAc 88:12); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.91 (d, *J* = 8 Hz, 1H), 7.52-7.50 (m, 1H), 7.46-7.43 (m, 3H), 7.35 (t, *J* = 7.5 Hz, 1H), 7.30 (d, *J* = 8 Hz, 2H), 4.26 (q, *J* = 7.25 Hz, 2H), 3.87 (s, 2H), 2.43 (s, 3H), 1.32 (t, *J* = 7.25 Hz, 3H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$): δ 168.8, 157.6, 157.1, 152.5, 147.1, 138.2, 130.7, 129.6, 129.1, 126.2, 124.4, 123.4, 120.9, 117.1, 112.6, 109.6, 61.7, 32.9, 21.3, 14.2 ppm; HRMS (ESI) *m/z* calcd. for $C_{22}H_{19}O_6$ $[M + H]^+$ 379.1176; found 379.1194.

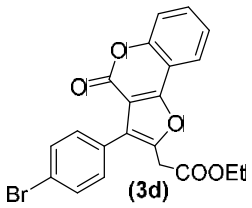
Ethyl 2-(3-(4-methoxyphenyl)-4-oxo-4*H*-furo[3,2-*c*]chromene-2-yl)acetate (3c)



(3c)

Yellow solid, m.p. 109.2 ºC, 67.6 mg, 83 % yield, (eluent: Petroleum ether: EtOAc 88:12); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.91 (dd, *J* = 7.5 Hz, *J* = 7.5 Hz, 1H), 7.53-7.44 (m, 4H), 7.37-7.34 (m, 1H), 7.03-7.01 (m, 2H), 4.26 (q, *J* = 7 Hz, 2H), 3.87 (s, 3H), 3.86 (s, 2H), 1.32 (t, *J* = 7 Hz, 3H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$): δ 168.8, 159.6, 157.1, 152.5, 146.9, 131.0, 130.7, 124.4, 123.2, 121.3, 120.9, 117.1, 113.9, 112.7, 109.6, 61.7, 55.3, 32.9, 14.2 ppm; HRMS (ESI) *m/z* calcd. for $C_{22}H_{18}NaO_5$ $[M + Na]^+$ 385.1046; found 385.1062.

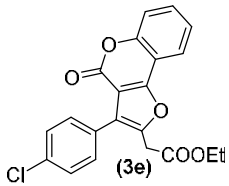
Ethyl 2-(3-(4-bromophenyl)-4-oxo-4*H*-furo[3,2-*c*]chromen-2-yl)acetate (3d)[3]



(3d)

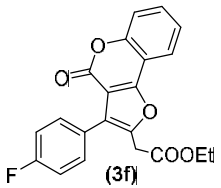
Yellow solid, m.p. 128 °C, 64 mg, 85% yield, (eluent: Petroleum ether: EtOAc 88:12); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.93 (dd, *J* = 8 Hz, *J* = 8 Hz, 1H), 7.63-7.61 (m, 2H), 7.56-7.54 (m, 1H), 7.47-7.43 (m, 3H), 7.40-7.36 (m, 1H), 4.26 (q, *J* = 7.25 Hz, 2H), 3.85 (s, 2H), 1.32 (t, *J* = 7 Hz, 3H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$): δ 168.5, 157.5, 157.3, 152.6, 147.4, 131.6, 131.4, 131.0, 128.1, 124.5, 122.8, 122.5, 121.0, 117.2, 112.5, 109.3, 51.9, 32.9, 14.1 ppm.

Ethyl 2-(3-(4-chlorophenyl)-4-oxo-4*H*-furo[3,2-*c*]chromen-2-yl)acetate (3e)[3]



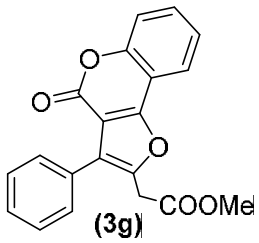
Yellow solid, m.p. 129.6 ^{o}C, 60.1mg, 78% yield, (eluent: Petroleum ether: EtOAc 88:12); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.92 (dd, *J* = 8 Hz, *J* = 8 Hz, 1H), 7.56 -7.53 (m, 1H), 7.50 – 7.44 (m, 5H), 7.38 - 7.35 (m, 1H), 4.25 (q, *J* = 7.25 Hz, 2H), 3.83 (s, 2H), 1.30 (t, *J*= 7.25 Hz, 3H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$): δ 168.5, 157.5, 157.3, 152.6, 147.4, 134.8, 131.1, 130.9, 129.0, 128.7, 128.0, 127.7, 124.5, 122.5, 121.0, 117.2, 112.5, 109.4, 61.8, 32.9, 14.1 ppm.

Ethyl 2-(3-(4-fluorophenyl)-4-oxo-4*H*-furo[3,2-*c*]chromen-2-yl)acetate (3f)



Brown solid, m.p. 73 ^{o}C, 66mg, 80 % yield, (eluent: Petroleum ether: EtOAc 88:12); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.94 (d, *J* = 8 Hz, 1H), 7.57-7.53 (m, 3H), 7.47 (d, *J* = 8 Hz, 1H), 7.38 (t, *J* = 7.5 Hz, 1H), 7.22 – 7.16 (m, 2H), 4.27 (q, *J* = 7.25 Hz, 2H), 3.85 (s, 2H), 1.32 (t, *J* = 7 Hz, 3H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$): δ 168.6, 157.2, 152.6, 147.3, 131.6, 131.5, 130.9, 125.2, 124.5, 122.6, 121.0, 117.2, 115.6, 115.4, 112.6, 61.8, 32.9, 29.7, 14.1 ppm.

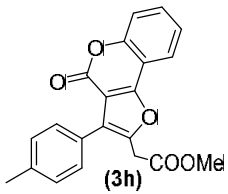
Methyl 2-(4-oxo-3-phenyl-4*H*-furo[3,2-*c*]chromene-2-yl)acetate (3g)



(3g)

Light yellow, m.p. 118.3 ^{o}C, 81.7mg, 93% yield, (eluent: Petroleum ether: EtOAc 88:12); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.94 (d, J = 8 Hz, 1H), 7.56-7.54 (m, 2H), 7.51-7.48 (m, 3H), 7.46-7.42 (m, 2H), 7.38 (t, J = 7.5 Hz, 1H), 3.89 (s, 2H), 3.81 (s, 3H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$): δ 169.1, 157.5, 157.2, 152.6, 147.1, 130.8, 129.8, 129.1, 128.5, 128.4, 124.4, 123.6, 121.0, 117.2, 112.6, 109.6, 52.7, 32.6 ppm. ESI *m/z* for $C_{20}H_{14}O_5$ [M + H]$^{+}$ found 335.0400.

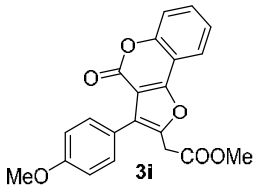
Methyl 2-(4-oxo-3-(*p*-tolyl)-4*H*-furo[3,2-*c*]chromene-2-yl)acetate (3h)



(3h)

Pale yellow solid, m.p. 129.7 ^{o}C, 81mg, 95 % yield, (eluent: Petroleum ether: EtOAc 88:12); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.93 (dd, J = 8 Hz, J = 7.5 Hz, 1H), 7.56-7.52 (m, 1H), 7.47-7.43 (m, 3H), 7.39-7.35 (m, 1H), 7.30 (d, J = 8 Hz, 2H), 3.88 (s, 2H), 3.81 (s, 3H), 2.43 (s, 3H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$): δ 169.2, 157.6, 157.2, 152.6, 146.9, 138.2, 130.7, 129.6, 129.2, 126.1, 124.3, 123.6, 121.0, 117.1, 112.7, 109.6, 52.7, 32.6, 21.3 ppm.

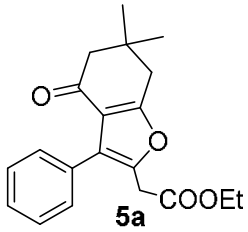
Methyl 2-(3-(4-methoxyphenyl)-4-oxo-4*H*-furo[3,2-*c*]chromen-2-yl)acetate (3i)



3i

Yellow solid, m.p.123.2 ^{o}C, 74.5mg, 90% yield, (eluent: Petroleum ether: EtOAc 88:12); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.93 (d, J = 7.5 Hz, 1H), 7.56-7.54 (m, 1H), 7.50-7.47 (m, 3H), 7.37 (t, J = 7.5 Hz, 1H), 7.02 (d, J = 8.5 Hz, 2H), 3.88 (s, 5H), 3.81 (s, 3H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$): δ 169.3, 159.6, 157.6, 157.1, 52.6, 146.7, 131.0, 130.7, 124.3, 123.3, 121.3, 121.0, 117.1, 113.9, 112.7, 109.6, 55.3, 52.7, 32.6 ppm.

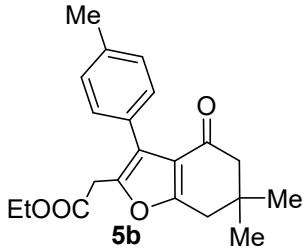
Ethyl 2-(6,6-dimethyl-4-oxo-3-phenyl-4,5,6,7-tetrahydrobenzofuran-2-yl)acetate (5a)[3]



5a

Yellow liquid (**59.4** mg, 78%). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.45 – 7.39 (m, 4H), 7.35 (d, *J* = 7 Hz, 1H), 4.22 (q, *J* = 7.25 Hz, 2H), 3.67 (s, 2H), 2.81 (s, 2H), 2.41 (s, 2H), 1.29 (t, *J* = 7 Hz, 3H), 1.19 (s, 6H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$): δ 193.4, 169.3, 165.8, 144.9, 134.6, 130.8, 129.7, 128.0, 127.6, 122.1, 61.4, 52.9, 37.6, 34.9, 32.5, 28.6, 14.1 ppm.

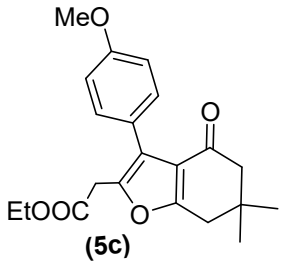
Ethyl 2-(6,6-dimethyl-4-oxo-3-(*p*-tolyl)-4,5,6,7-tetrahydrobenzofuran-2-yl)acetate (5b)[2]



5b

Yellow liquid, 62.4 mg, 80% yield, (eluent: Petroleum ether: EtOAc 80:20); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.33 (d, *J* = 7.5 Hz, 2H), 7.12 (d, *J* = 7.5 Hz, 2H), 4.22 (q, *J* = 7 Hz, 2H), 3.69 (s, 2H), 2.80 (s, 2H), 2.40 (s, 2H), 2.39 (s, 3H), 1.19 (s, 6H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$): δ 193.4, 169.4, 165.8, 144.7, 137.3, 129.5, 128.7, 127.7, 122.0, 118.6, 61.4, 52.99, 37.6, 34.8, 32.5, 28.6, 21.3, 14.1 ppm.

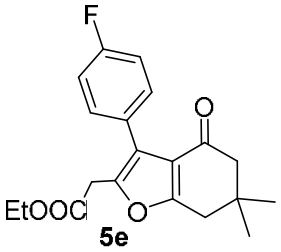
Ethyl 2-(3-(4-methoxyphenyl)-6,6-dimethyl-4-oxo-4,5,6,7-tetrahydrobenzofuran-2-yl) acetate (5c)



(5c)

Yellow liquid, 72.5 mg, 89% yield; (eluent: Petroleum ether: EtOAc 80:20); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.37 (d, *J* = 8.5 Hz, 2H), 6.94 (d, *J* = 8 Hz, 2H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$): δ 193.5, 169.4, 165.8, 159.0, 144.5, 130.9, 123.0, 121.7, 118.5, 113.5, 61.4, 55.2, 52.9, 37.6, 34.8, 32.5, 28.6, 14.1 ppm; HRMS (ESI) *m/z* calcd. for $C_{21}H_{24}O_5$ $[M + H]^+$ 365.1364; found 365.1348.

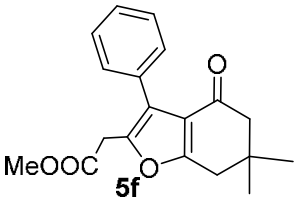
Ethyl 2-(3-(4-fluorophenyl)-6,6-dimethyl-4-oxo-4,5,6,7-tetrahydrobenzofuran-2-yl)acetate (5e)



5e

Yellow solid, m.p. 65.6 ^{o}C, 58.1mg, 75% yield; (eluent: Petroleum ether: EtOAc 80:20); ^{1}H NMR (5100 MHz, $CDCl_3$): δ 7.42 (q, *J* = 9 Hz, 2H), 7.09 (t, *J* = 8.5 Hz, 2H), 4.22 (q, *J* = 7 Hz, 2H), 3.64 (s, 2H), 2.80 (s, 2H), 2.40 (s, 2H), 1.29 (t, *J* = 7 Hz, 3H), 1.18 (s, 6H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$): δ 193.5, 169.2, 165.9, 161.4, 144.9, 131.5, 131.4, 126.7, 121.2, 118.4, 115.1, 114.9, 61.5, 52.9, 37.6, 34.9, 32.4, 28.5, 14.1 ppm.

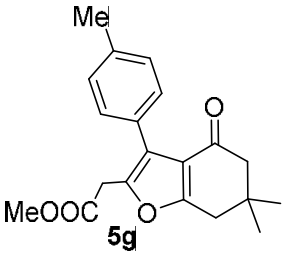
Methyl 2-(6,6-dimethyl-4-oxo-3-phenyl-4,5,6,7-tetrahydrobenzofuran-2-yl)acetate (5f)



5f

Brown solid, m.p.81.3 ^{o}C, 70.6 mg, 86% yield; (eluent: Petroleum ether: EtOAc 80:20); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.45-7.39 (m, 4H), 7.36-7.35 (m, 1H), 3.77 (s, 3H), 3.69 (s, 2H), 2.81 (s, 2H), 2.41 (s, 2H), 1.19 (s, 6H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ 193.4, 169.8, 165.9, 144.7, 129.7, 128.0, 128.1, 127.66, 122.2, 118.5, 52.9, 52.5, 37.6, 34.9, 32.2, 28.6 ppm.

Methyl 2-(6,6-dimethyl-4-oxo-3-(*p*-tolyl)-4,5,6,7-tetrahydrobenzofuran-2-yl)acetate (5g)

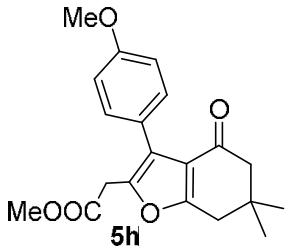


5g

Yellow liquid, 59.9 mg, 75% yield; (eluent: Petroleum ether: EtOAc 80:20); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.32 (d, *J* = 8 Hz, 2H), 7.22 (d, *J* = 8 Hz, 2H), 3.76 (s, 3H), 3.68 (s, 2H), 2.80 (s, 2H), 2.40 (s, 2H), 2.39 (s, 3H), 1.19 (s, 6H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ 193.4, 169.9, 165.8, 144.5, 137.3, 129.5, 129.3, 128.8, 127.7, 122.1, 118.5, 52.9, 52.4, 37.6, 34.8, 32.2, 28.6, 21.3 ppm; ESI *m/z* for $C_{20}H_{22}O_4$ $[M + H]^+$;

found 327.1113.

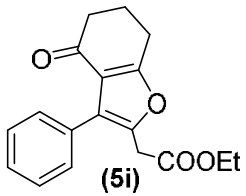
Methyl 2-(3-(4-methoxyphenyl)-6,6-dimethyl-4-oxo-4,5,6,7-tetrahydrobenzofuran-2-yl)acetate (5h)



5h

Yellow liquid; 64.5 mg, 83% yield; (eluent: Petroleum ether: EtOAc 80:20); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.37 (d, *J* = 9 Hz, 2H), 6.94 (d, *J* = 8.5 Hz, 2H), 3.85 (s, 3H), 3.77 (s, 3H), 3.67 (s, 2H), 2.79 (s, 2H), 2.40 (s, 2H), 1.18 (s, 6H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ 193.5, 169.9, 165.8, 159.1, 144.3, 130.8, 122.9, 121.8, 118.5, 113.5, 55.2, 52.9, 52.4, 37.6, 34.8, 32.9, 32.2, 28.6 ppm.

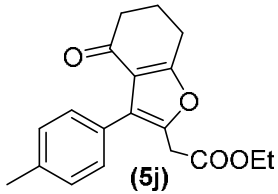
Ethyl 2-(4-oxo-3-phenyl-4,5,6,7-tetrahydrobenzofuran-2-yl)acetate (5i)[3]



(5i)

Brown liquid, 51.5 mg, 74% yield; (eluent: Petroleum ether: EtOAc 80:20); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.42 (d, *J* = 1 Hz, 2H), 7.40 (d, *J* = 1.5 Hz, 1 H), 7.42-7.31 (m, 2H), 4.24 - 4.20 (m, 2H), 3.66 (s, 2H), 2.94 (t, *J* = 6.5 Hz, 2H), 2.52 (t, *J* = 6.5 Hz, 2H), 2.22 (t, *J* = 6.5 Hz, 2H), 1.30 (t, *J* = 7 Hz, 3H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ 193.9, 169.4, 166.7, 144.6, 130.88, 129.7, 128.7, 128.6, 128.0, 127.6, 122.3, 119.7, 61.4, 38.6, 32.4, 23.7, 22.3, 14.1 ppm.

Ethyl 2-(4-oxo-3-(*p*-tolyl)-4,5,6,7-tetrahydrobenzofuran-2-yl)acetate (5j)[2]

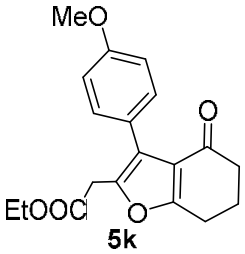


(5j)

Brown liquid, 57.9 mg, 81% yield; (eluent: Petroleum ether: EtOAc 80:20); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.32 – 7.30 (m, 2H), 7.21 (d, *J* = 8 Hz, 2H), 4.22 (q, *J* = 7.25 Hz, 2H), 3.65 (s, 2H), 2.93 (t, *J* = 6.5 Hz, 2H), 2.52 (t, *J* = 6.5 Hz, 2H), 2.40 (s, 3H), 2.21 (quint, *J* = 6.5 Hz, 2H), 1.30 (t, *J* = 7 Hz, 3H) ppm. ^{13}C NMR (125

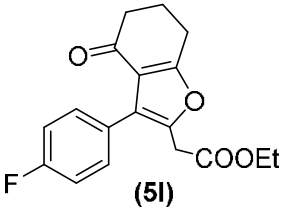
MHz, $CDCl_3$): δ 193.9, 169.4, 166.6, 144.4, 137.4, 129.5, 128.7, 127.8, 122.2, 119.8, 61.4, 38.6, 32.4, 23.7, 22.4, 21.3, 14.1 ppm.

Ethyl 2-(3-(4-methylphenyl)-4-oxo-4,5,6,7-tetrahydrobenzofuran-2-yl)acetate (5k)



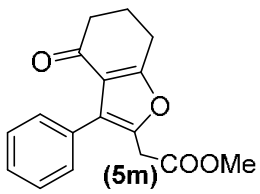
Brown liquid, 51.6 mg, 73 % yield; (eluent: Petroleum ether: EtOAc 80:20); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.34 (d, *J* = 9 Hz, 2H), 6.92 (d, *J* = 9 Hz, 2H), 4.20 (q, *J* = 7.25 Hz, 2H), 3.83(s, 3H), 3.63 (s, 2H), 2.91 (t, *J* = 6 Hz, 2H), 2.49 (t, *J* = 7 Hz, 2H), 2.19 (t, *J* = 6.5 Hz, 2H), 1.29-1.25 (m, 3H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$): δ 194.0, 169.5, 166.6, 159.0, 144.2, 130.8, 123.1, 121.9, 119.7, 113.5, 61.4, 55.2, 38.6, 32.4, 23.7, 22.4, 14.1 ppm; HRMS (ESI) *m/z* calcd. for $C_{19}H_{20}O_5$ $[M + Na]^+$ 351.1208; found 351.1262.

Ethyl 2-(3-(4-fluorophenyl)-4-oxo-4,5,6,7-tetrahydrobenzofuran-2-yl)acetate (5l)



Yellow liquid, 62.2 mg, 87% yield; (eluent: Petroleum ether: EtOAc 80:20); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.42 – 7.39 (m, 2H), 7.11-7.07 (m, 2H), 4.22 (q, *J* = 7.25 Hz, 2H), 3.63 (s, 2H), 2.94 (t, *J* = 6.5 Hz, 2H), 2.52 (t, *J* = 7 Hz, 2H), 2.21 (t, *J* = 6.5 Hz, 2H), 1.30 (t, *J* = 7.5 Hz, 3H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$): δ 194.0, 169.3, 166.7, 144.6, 131.4, 131.3, 121.4, 119.6, 115.1, 114.9, 61.5, 38.5, 32.3, 23.7, 22.3, 14.1 ppm; HRMS (ESI) *m/z* calcd. for $C_{18}H_{17}FO_4$ $[M + Na]^+$ 367.1321; found 367.1306.

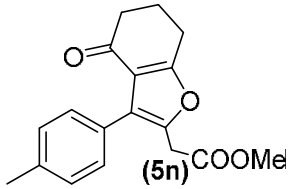
Methyl 2-(4-oxo-3-phenyl-4,5,6,7-tetrahydrobenzofuran-2-yl)acetate (5m)



Brown liquid, 65.7 mg, 88% yield; (eluent: Petroleum ether: EtOAc 80:20); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.42 – 7.40 (m, 3H), 7.39 – 7.30 (m, 2H), 3.77 (s, 3H), 3.68 (s, 2H), 2.94 (t, *J* = 6.25 Hz, 2H), 2.52 (t, *J* = 6.5 Hz, 2H), 2.22 (quint, *J* = 6.5 Hz, 2H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$): δ 193.8, 169.8, 166.7, 144.4, 130.8,

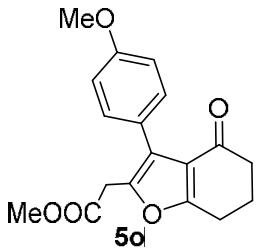
129.6, 128.0, 127.6, 122.3, 119.7, 52.5, 38.6, 32.1, 23.7, 22.3 ppm; HRMS (ESI) *m/z* calcd. for $C_{17}H_{16}O_4$ $[M + H]^+$ 307.0946; found 307.0931.

Methyl 2-(4-oxo-3-phenyl-4,5,6,7-tetrahydrobenzofuran-2-yl)acetate (5n)



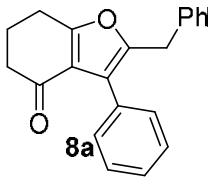
Brown solid, m.p. 69 ^oC, 62.1 mg, 85% yield; (eluent: Petroleum ether: EtOAc 80:20) ; ^1H NMR (500 MHz, $CDCl_3$): δ 7.31 (m, 2H), 7.21(d, *J* = 8 Hz, 2H), 3.76 (s, 3H), 3.67 (s, 2H), 2.93 (t, *J* = 6 Hz, 2H), 2.52 (t, *J* = 7.5 Hz, 2H), 2.39 (s, 3H), 2.21 (quint, *J* = 6.5 Hz, 2H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$): δ 193.9, 169.9, 166.7, 144.2, 137.3, 129.5, 128.8, 127.7, 122.3, 119.8, 52.4, 38.6, 32.1, 23.7, 22.3, 21.3 ppm. ESI *m/z* $C_{18}H_{18}O_4$ $[M + H]^+$ 298.1205.

Methyl 2-(3-(4-methoxyphenyl)-4-oxo-4,5,6,7-tetrahydrobenzofuran-2-yl)acetate (5o)



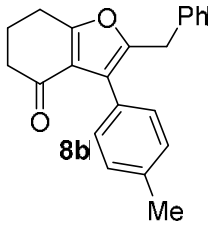
Brown solid, m.p. 83.4 ^oC, 62.8 mg, 88% yield; (eluent: Petroleum ether: EtOAc 80:20); ^1H NMR (500 MHz, $CDCl_3$): δ 7.33 (d, *J* = 8.5 Hz, 2H), 6.93 (d, *J* = 9 Hz, 2H), 3.83 (s, 3H), 3.75 (s, 3H), 3.65 (s, 2H), 2.91 (t, *J* = 6 Hz, 2H), 2.50 (t, *J* = 7 Hz, 2H), 2.19 (t, *J* = 6 Hz, 2H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$): δ 194.0, 169.9, 166.7, 159.1, 144.0, 130.8, 123.0, 122.0, 119.8, 113.5, 55.2, 52.5, 38.6, 32.2, 23.7, 22.3 ppm.

2-benzyl-3-phenyl-6,7-dihydrobenzofuran-4(5*H*)-one (8a)[4]



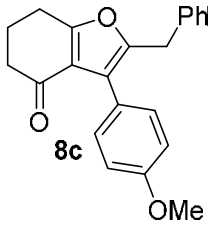
Yellow solid, m.p. 73.4 °C, 65.3 mg, 90% yield, (eluent: Petroleum ether: EtOAc 90:10); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.43 -7.40 (m, 4H), 7.37 - 7.33 (m, 3H), 7.28 (d, *J* = 7.5 Hz, 1H), 7.23 (d, *J* = 7.5 Hz, 2H), 4.01 (s, 2H), 2.90 (t, *J* = 6.5 Hz, 2H), 2.52 (t, *J* = 6 Hz, 2H), 2.20 (t, *J* = 6.5 Hz, 2H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) : δ 194.1, 166.5, 150.4, 137.9, 131.5, 129.8, 128.6, 128.4, 128, 127.4, 126.6, 120.5, 119.7, 38.6, 32, 23.7, 22.4 ppm.

2-benzyl-3-(*p*-tolyl)-6,7-dihydrobenzofuran-4(5*H*)-one (8b)



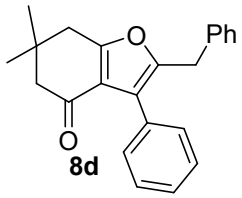
Brown liquid, 61.2 mg, 86% yield, (eluent: Petroleum ether: EtOAc 90:10); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.36 - 7.32 (m, 4H), 7.28 - 7.26 (m, 1H), 7.22 (d, *J* = 7.5 Hz, 2H), 6.95 (d, *J* = 8.5 Hz, 2H), 3.99 (s, 2H), 3.86 (s, 2H), 2.89 (t, *J* = 6 Hz, 3H), 2.51 (t, *J* = 6 Hz, 2H), 2.19 (t, *J* = 6 Hz, 2H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) : δ 194.2, 166.4, 158.9, 150, 138.1, 130.9, 128.6, 128.4, 126.6, 123.7, 120, 119.8, 113.5, 55.2, 38.6, 32, 23.8, 22.4 ppm.

2-benzyl-3-(4-methoxyphenyl)-6,7-dihydrobenzofuran-4(5*H*)-one (8c)



Brown liquid, 62.7 mg, 90%yield, (eluent: Petroleum ether: EtOAc 90:10); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.36 - 7.33 (m, 4H), 7.29 - 7.27 (m, 1H), 7.23 (s, 4H), 4.01 (s, 2H), 2.90 (t, *J* = 6.5 Hz, 2H), 2.52 (t, *J* = 6 Hz, 2H), 2.42 (s, 3H), 2.20 (t, *J* = 6.5 Hz, 2H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) : δ 194., 166.4, 150.2, 138.1, 137, 129.6, 128.7, 128.6, 128.5, 128.4, 126.6, 120.4, 119.8, 38.6, 32, 23.8, 22.5, 21.3 ppm.

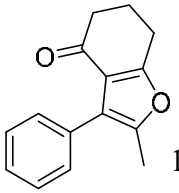
2-benzyl-6,6-dimethyl-3-phenyl-6,7-dihydrobenzofuran-4(5*H*)-one (8d)



8d

Yellow solid, m.p. 59.3 ^{o}C, 72.2 mg, 91% yield, (eluent: Petroleum ether: EtOAc 90:10); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.44 - 7.39 (m, 5H), 7.36 - 7.32 (m, 3H), 7.22 (d, *J* = 7.5 Hz, 2H), 4.02 (s, 2H), 2.77 (s, 2H), 2.41 (s, 2H), 1.18 (s, 6H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$) : δ 193.7, 165.7, 150, 138, 131.3, 129.7, 128.6, 128.4, 128, 127.4, 126.6, 124.4, 120.4, 118.5, 52.9, 37.7, 34.9, 32.1, 28.6 ppm.

2-methyl-3-phenyl-6,7-dihydrobenzofuran-4(5H)-one (10a)[5]

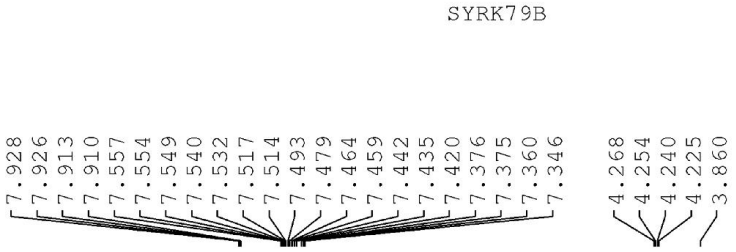


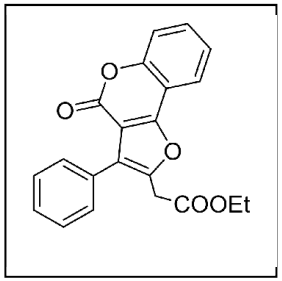
Yellow liquid; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.41 (d, *J* = 4 Hz, 4H), 7.35 - 7.32 (m, 1H), 2.91 (t, *J* = 6.5 Hz, 2H), 2.52 (t, *J* = 6 Hz, 2H), 2.34 (s, 3H), 2.21 (m, 2H), ppm; ^{13}C NMR (125 MHz, $CDCl_3$): δ 194.2, 165.8, 148.8, 131.7, 129.8, 127.9, 127.1, 119.7, 119.2, 38.7, 23.7, 22.6, 12.1; HRMS (EI): m/z calculated for C15H14O2 226.09883 found 226.09839.

6- References

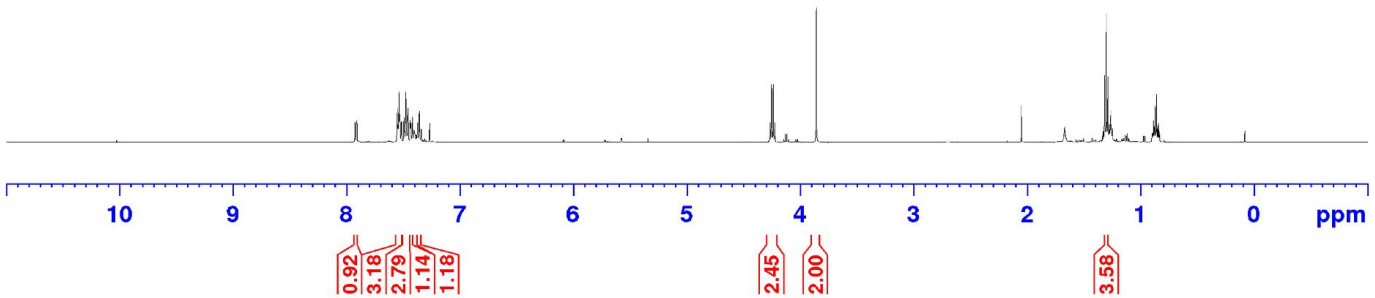
1. (a) C. Guo, X. Lu, *J. Chem. Soc. Chem. Comm.*, 1993, 394-395; (b) S. Yaragorla, R. Muthyala, *Tetrahedron Lett*, 2010, **51**, 467-470.
2. D. K. Nair, M. M. Saikh and N.N.N. Irish, *Tetrahedron,* 2012**, 53**, 3349.
3. W. Y. Huang, Y. C. Chen and K. Chen, *Chem. Asian. J.*, 2012, 688
4. S. Porna, M. Gohain, J. Tonder and B. Bezuidenhouldt, *Synlett.,* 2015**, 26***,* 745-750.
5. V. Cadierno, J. Díez, J. Gimeno and N. Nebra. *J. Org. Chem.*, 2008, **73**, 5852.

3a SYRK79B

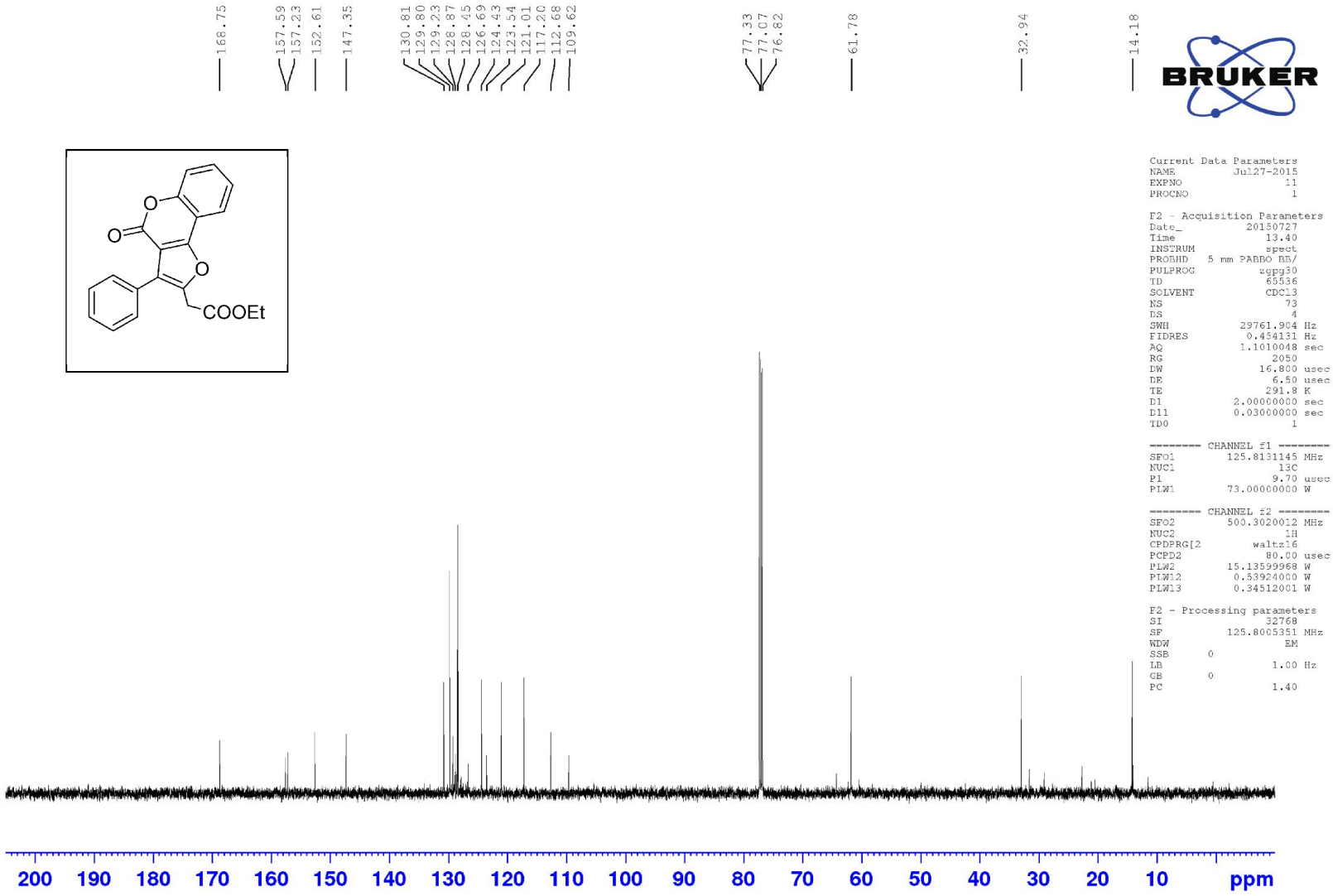




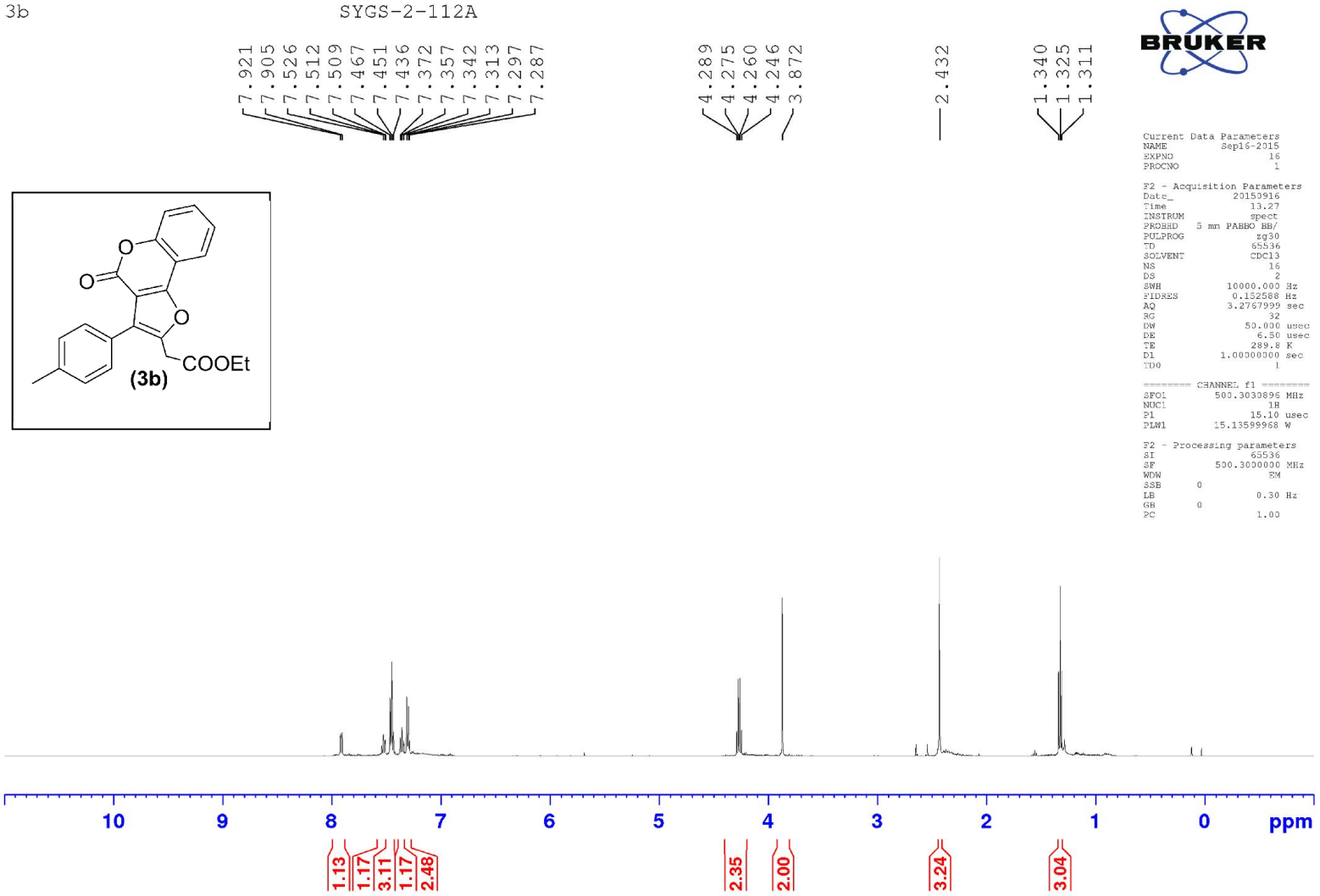

```
Current Data Parameters
NAME                  Jul27-2015
EXPNO                         10
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20150727
Time                       13.35
INSTRUM                    spect
PROBHD    5 mm PABBO BB/
PULPROG                     zg30
TD                         65536
SOLVENT                    CDCl3
NS                            14
DS                             2
SWH                   10000.000 Hz
FIDRES                0.152588 Hz
AQ                    3.2767999 sec
RG                         90.5
DW                       50.000 usec
DE                        6.50 usec
TE                       290.4 K
D1                    1.00000000 sec
TD0                            1

======== CHANNEL f1 ========
SFO1                500.3030896 MHz
NUC1                        1H
P1                       15.10 usec
PLW1                15.13599968 W

F2 - Processing parameters
SI                         65536
SF                  500.3000092 MHz
WDW                           EM
SSB                0
LB                        0.30 Hz
GB                 0
PC                         1.00
```



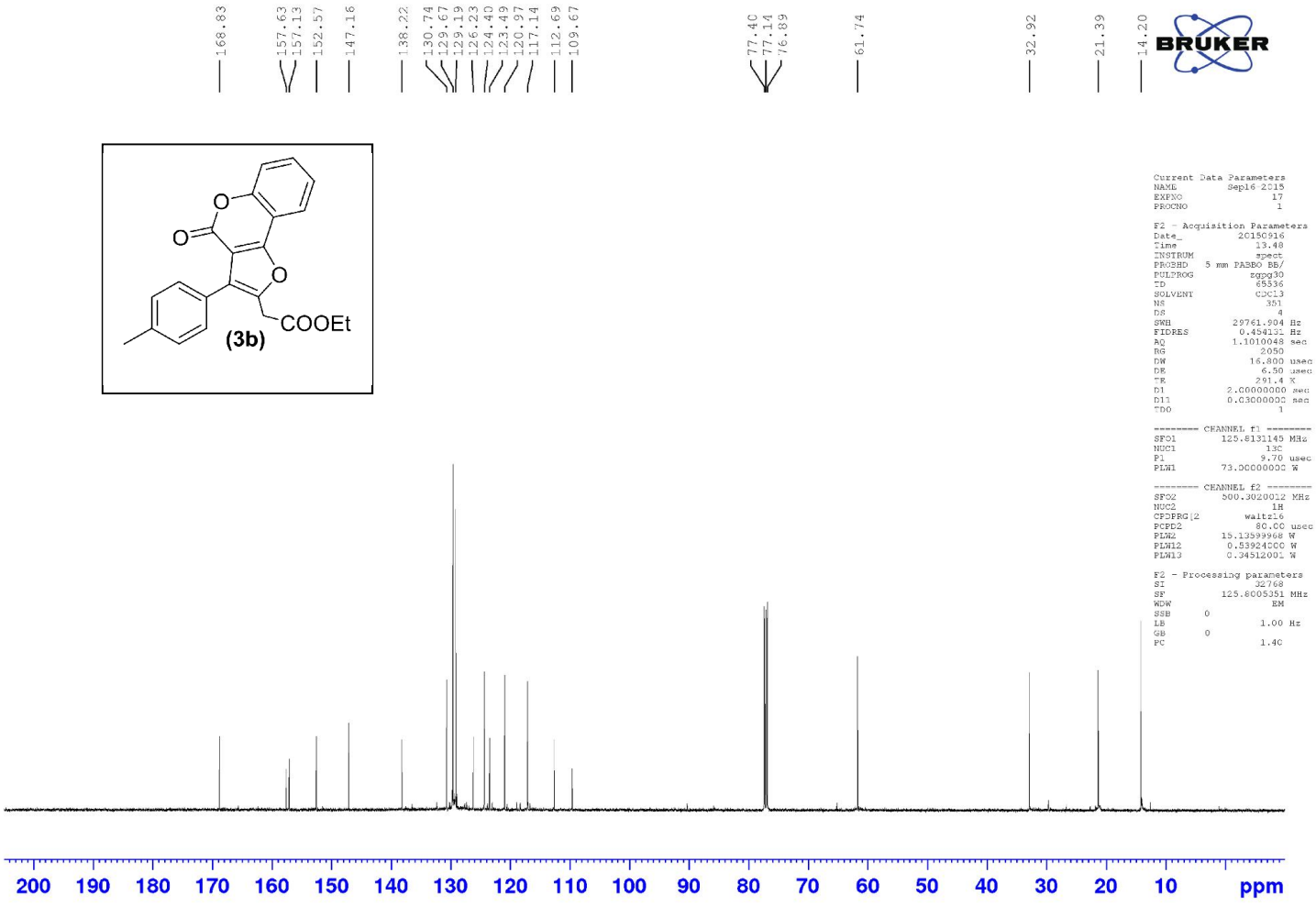
SYRK79B



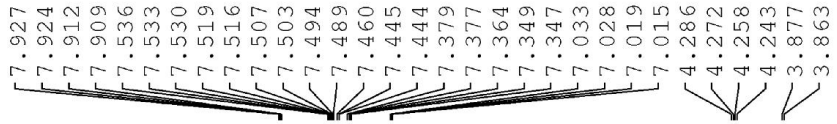

```
Current Data Parameters
NAME                  Jul27-2015
EXPNO                         11
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20150727
Time                       13.40
INSTRUM                    spect
PROBHD    5 mm PABBO BB/
PULPROG                 zgpg30
TD                         65536
SOLVENT                    CDCl3
NS                            73
DS                             4
SWH                    29761.904 Hz
FIDRES                 0.454131 Hz
AQ                     1.1010048 sec
RG                          2050
DW                        16.800 usec
DE                         6.50 usec
TE                        291.8 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                            1

======== CHANNEL f1 ========
SFO1                125.8131145 MHz
NUC1                        13C
P1                         9.70 usec
PLW1                73.00000000 W

======== CHANNEL f2 ========
SFO2                500.3020012 MHz
NUC2                        1H
CPDPRG[2                waltz16
PCPD2                     80.00 usec
PLW2                15.13599968 W
PLW12                0.53924000 W
PLW13                0.34512001 W

F2 - Processing parameters
SI                         32768
SF                  125.8005351 MHz
WDW                           EM
SSB                0
LB                         1.00 Hz
GB                 0
PC                         1.40
```

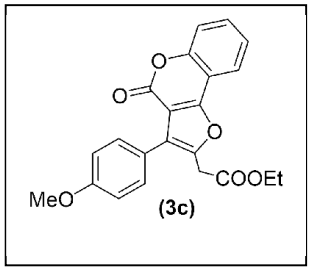

3b
SYGS-2-112A
7.921
7.905
7.526
7.512
7.509
7.467
7.451
7.436
7.372
7.357
7.342
7.313
7.297
7.287
4.289
4.275
4.260
4.246
3.872
2.432
1.340
1.325
1.311
BRUKER
Current Data Parameters
NAME Sep16-2015
EXPNO 16
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150916
Time 13.27
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 32
DW 50.000 usec
DE 6.50 usec
TE 289.8 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
SFO1 500.3030896 MHz
NUC1 1H
P1 15.10 usec
PLW1 15.13599968 W
F2 - Processing parameters
SI 65536
SF 500.3000000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
O
O
COOEt
(3b)
10
9
8
7
6
5
4
3
2
1
0
ppm
1.13
1.17
3.11
1.17
2.48
2.35
2.00
3.24
3.04

SYGS-2-112A

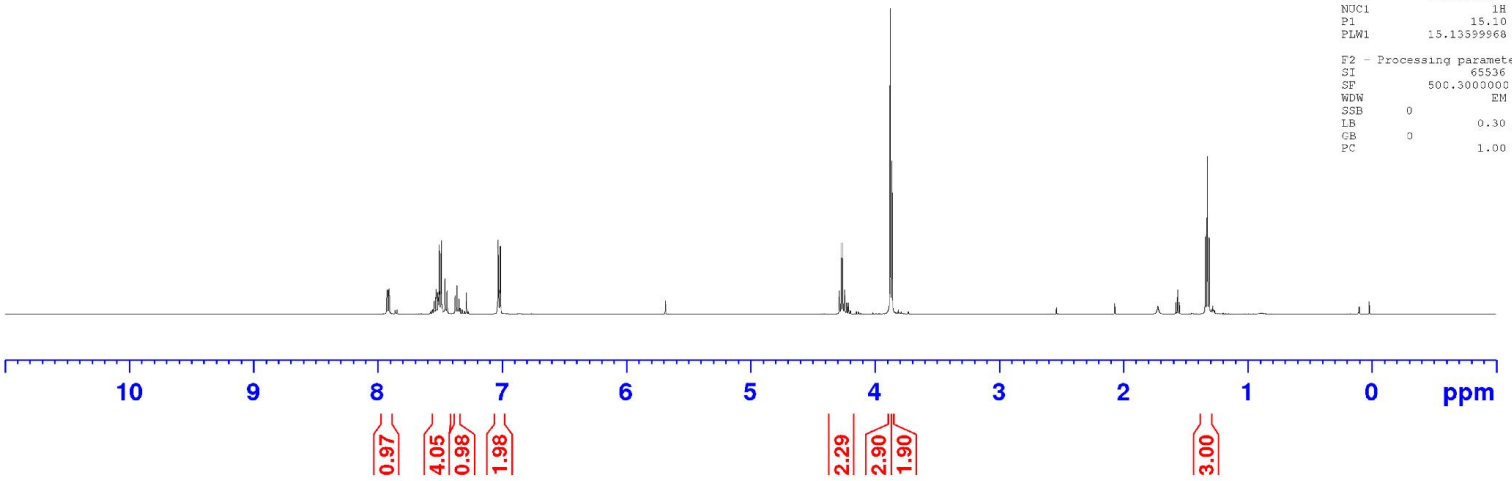

168.83
157.63
157.13
152.57
147.16
138.22
130.74
129.67
129.19
126.23
124.40
123.49
120.97
117.14
112.69
109.67
77.40
77.14
76.89
61.74
32.92
21.39
14.20
BRUKER
O
O
O
COOEt
(3b)
Current Data Parameters
NAME Sep16 2015
EXPNO 17
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150916
Time 13.48
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 351
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 291.4 K
D1 2.00000000 sec
D11 0.03000002 sec
TD0 1
CHANNEL f1
SFO1 125.8133145 MHz
NUC1 13C
P1 9.70 usec
PLW1 73.00000002 W
CHANNEL f2
SFO2 500.3020012 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 15.13599968 W
PLW12 0.53924000 W
PLW13 0.34512001 W
F2 - Processing parameters
SI 32768
SF 125.8005351 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

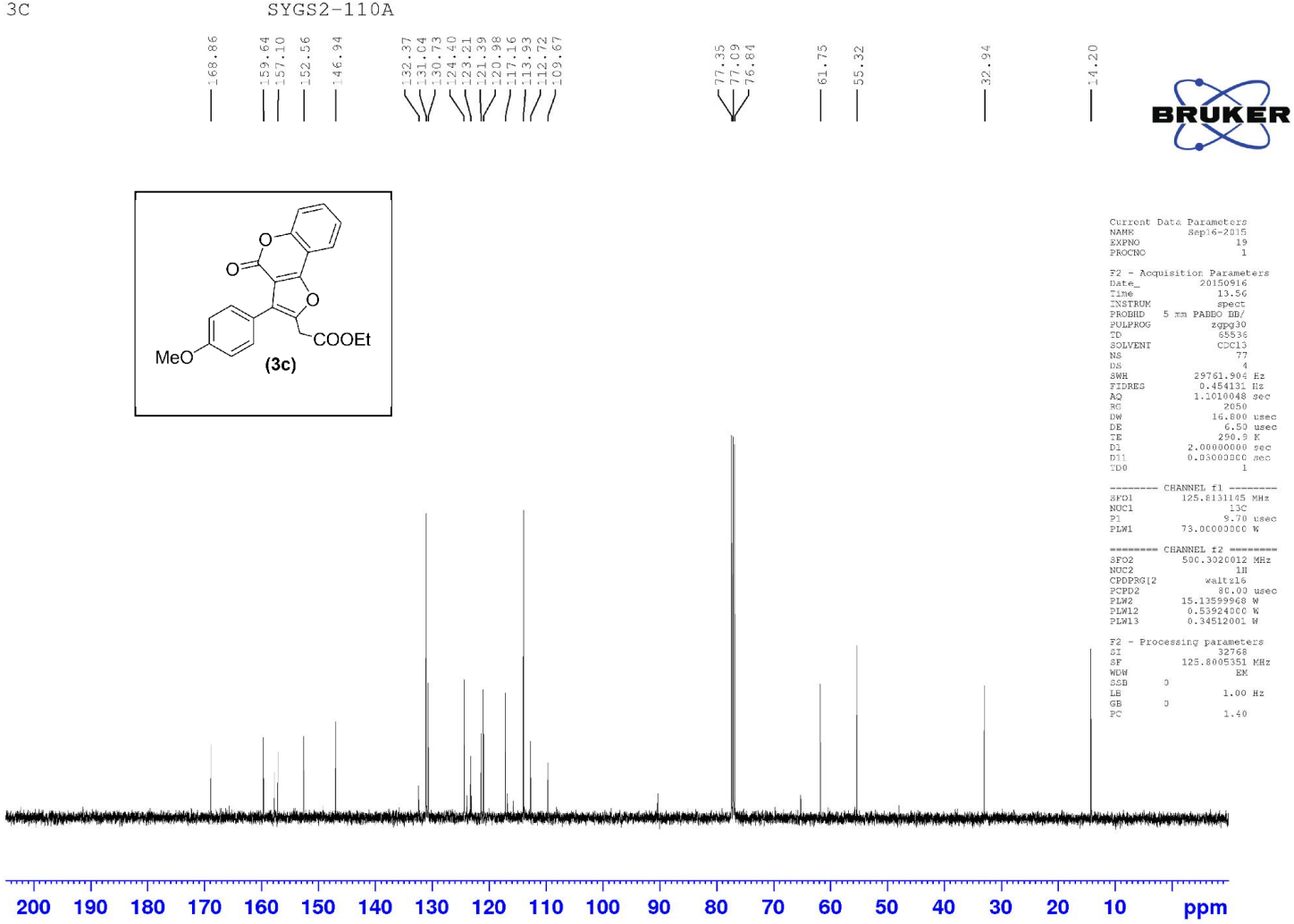
3c SYGS2-110A






```
Current Data Parameters
NAME                Sep16-2015
EXPNO                       16
PROCNO                       1

F2 - Acquisition Parameters
Date_               20150916
Time                    13.52
INSTRUM                 spect
PROBHD    5 mm PABBO BB/
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                          9
DS                          2
SWH                10000.000 Hz
FIDRES                 0.152588 Hz
AQ                 3.2767999 sec
RG                       80.6
DW                     50.000 usec
DE                      6.50 usec
TE                     289.9 K
D1                 1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1               500.3030896 MHz
NUC1                       1H
P1                      15.10 usec
PLW1               15.13599968 W

F2 - Processing parameters
SI                     65536
SF                 500.3000000 MHz
WDW                       EM
SSB          0
LB                      0.30 Hz
GB           0
PC                      1.00
```



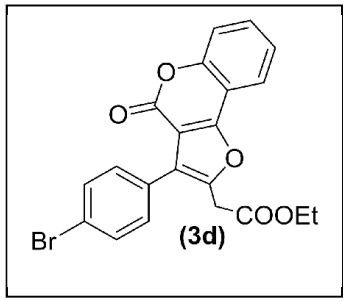

3C
SYGS2-110A
168.86
159.64
157.10
152.56
146.94
132.37
131.04
130.73
124.40
123.21
121.39
120.98
117.16
113.93
112.72
109.67
77.35
77.09
76.84
61.75
55.32
32.94
14.20
BRUKER
O
O
O
COOEt
MeO
(3c)
Current Data Parameters
NAME Sep16-2015
EXPNO 19
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150916
Time 13.56
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 77
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 290.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
SFO1 125.8131145 MHz
NUC1 13C
P1 9.70 usec
PLW1 73.00000000 W
CHANNEL f2
SFO2 500.3020012 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 15.13599968 W
PLW12 0.53924000 W
PLW13 0.34512001 W
F2 - Processing parameters
SI 32768
SF 125.8005351 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

3d SYGS2-110

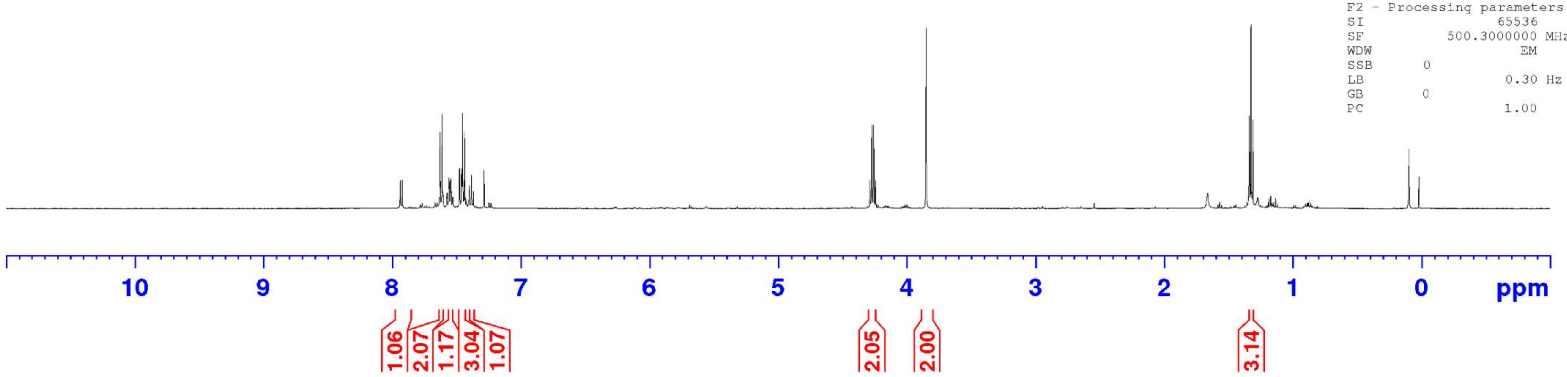
7.942 7.939 7.926 7.923 7.630 7.627 7.617 7.613 7.563 7.561 7.558 7.547 7.543 7.478 7.461 7.456 7.452 7.443 7.439 7.402 7.400 7.386 7.371 7.369

4.288 4.273 4.259 4.245 3.851

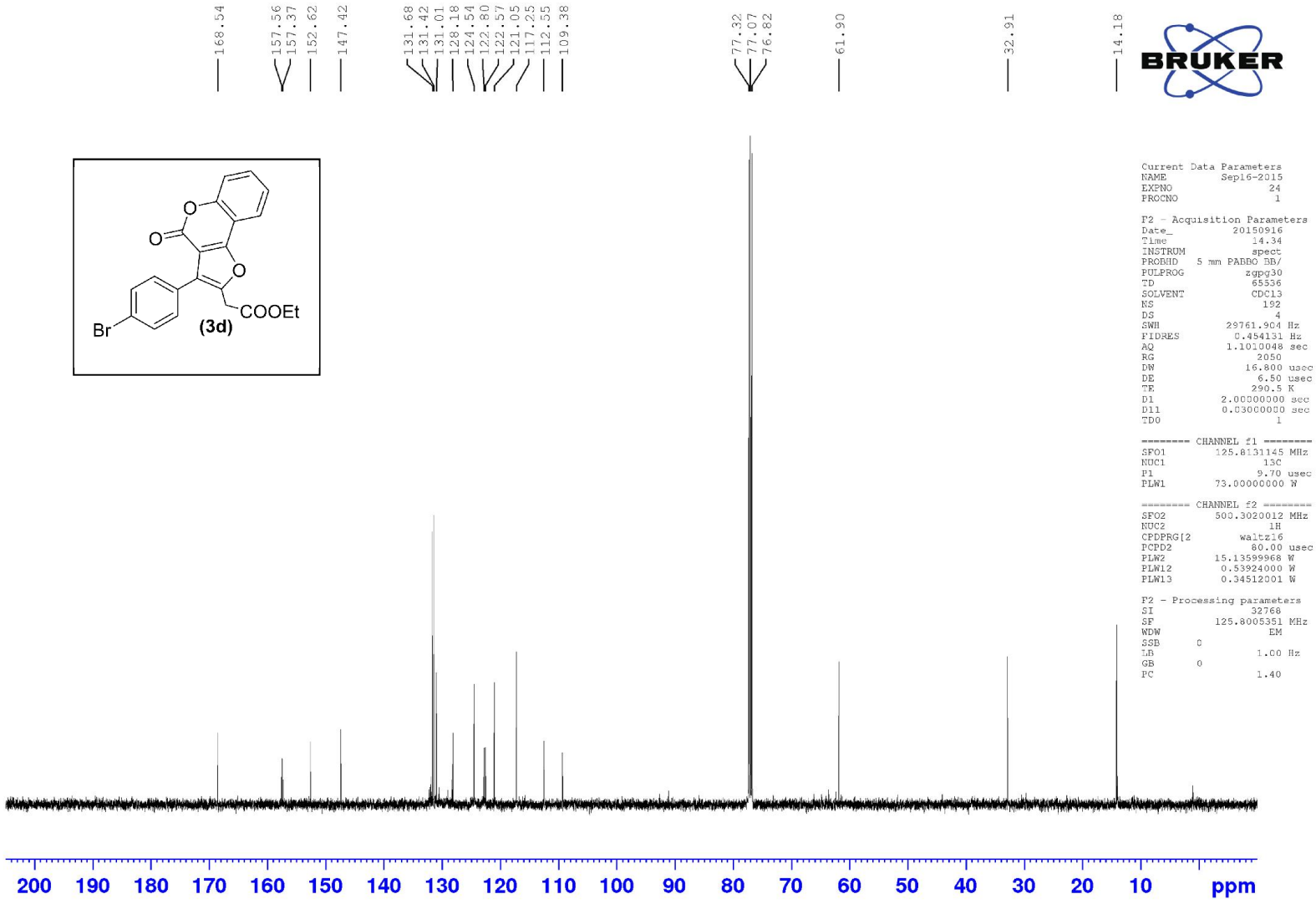
1.340 1.326 1.312



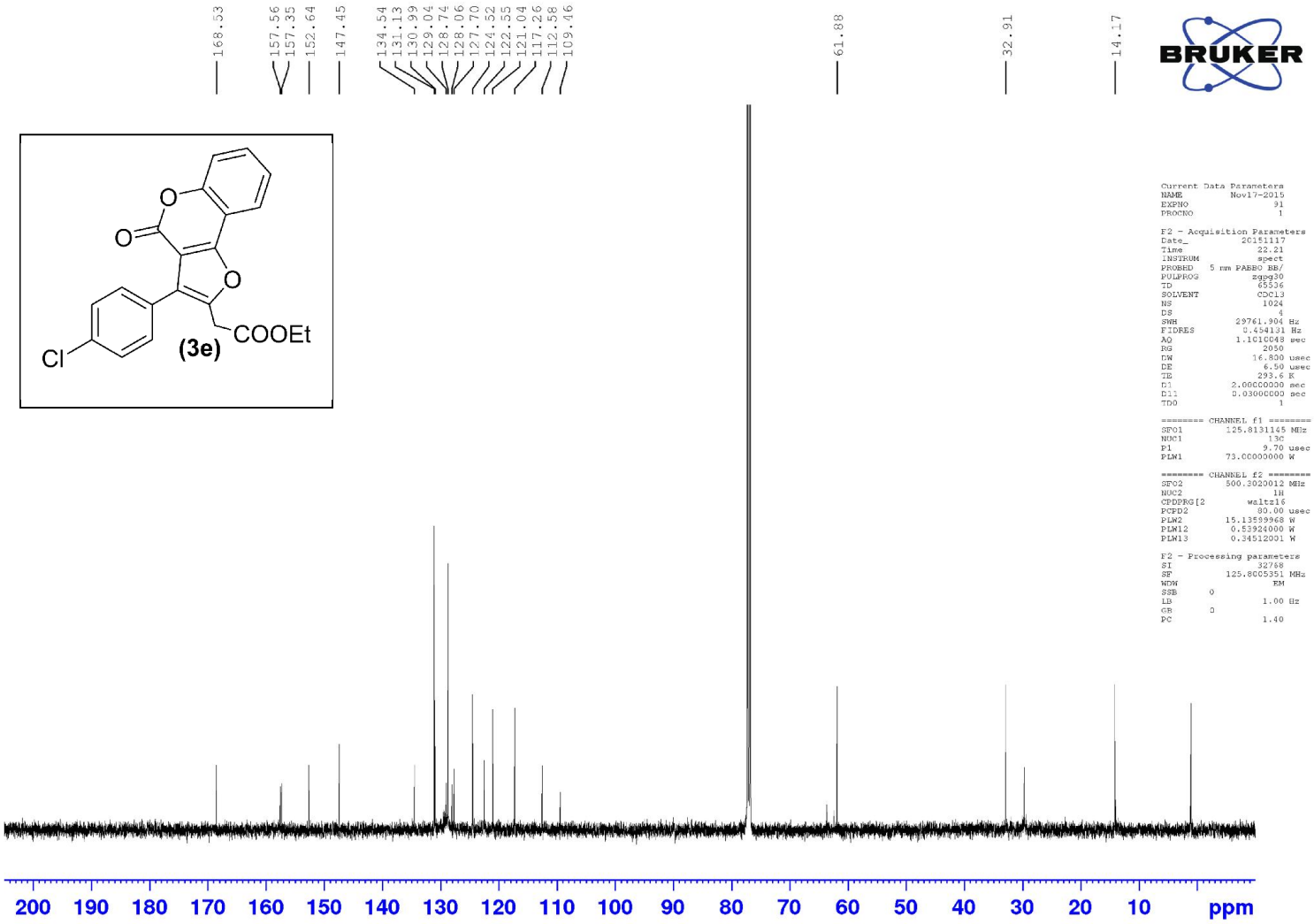

```
Current Data Parameters
NAME                  Sep16-2015
EXPNO                         23
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20150916
Time                       14.31
INSTRUM                    spect
PROBHD    5 mm PABBO BB/
PULPROG                    zg30
TD                        65536
SOLVENT                   CDCl3
NS                           16
DS                            2
SWH                   10000.000 Hz
FIDRES                0.152588 Hz
AQ                    3.2767999 sec
RG                          128
DW                       50.000 usec
DE                        6.50 usec
TE                       289.7 K
D1                    1.00000000 sec
TD0                            1

======== CHANNEL f1 ========
SFO1                500.3030896 MHz
NUC1                        1H
P1                       15.10 usec
PLW1                15.13599968 W

F2 - Processing parameters
SI                        65536
SF                  500.3000000 MHz
WDW                          EM
SSB                   0
LB                        0.30 Hz
GB                    0
PC                        1.00
```



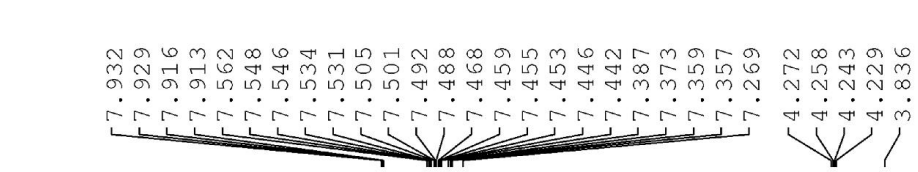
3d SYGS2-110

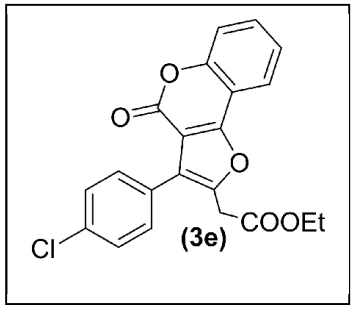

168.54
157.56
157.37
152.62
147.42
131.68
131.42
131.01
128.18
124.54
122.80
122.57
121.05
117.25
112.55
109.38
77.32
77.07
76.82
61.90
32.91
14.18
BRUKER
O
O
O
Br
COOEt
(3d)
Current Data Parameters
NAME Sep16-2015
EXPNO 24
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150916
Time 14.34
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 192
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 290.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
SFO1 125.8131145 MHz
NUC1 13C
P1 9.70 usec
PLW1 73.00000000 W
CHANNEL f2
SFO2 500.3020012 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 15.13599968 W
PLW12 0.53924000 W
PLW13 0.34512001 W
F2 - Processing parameters
SI 32768
SF 125.8005351 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

3e SY-GS2-112

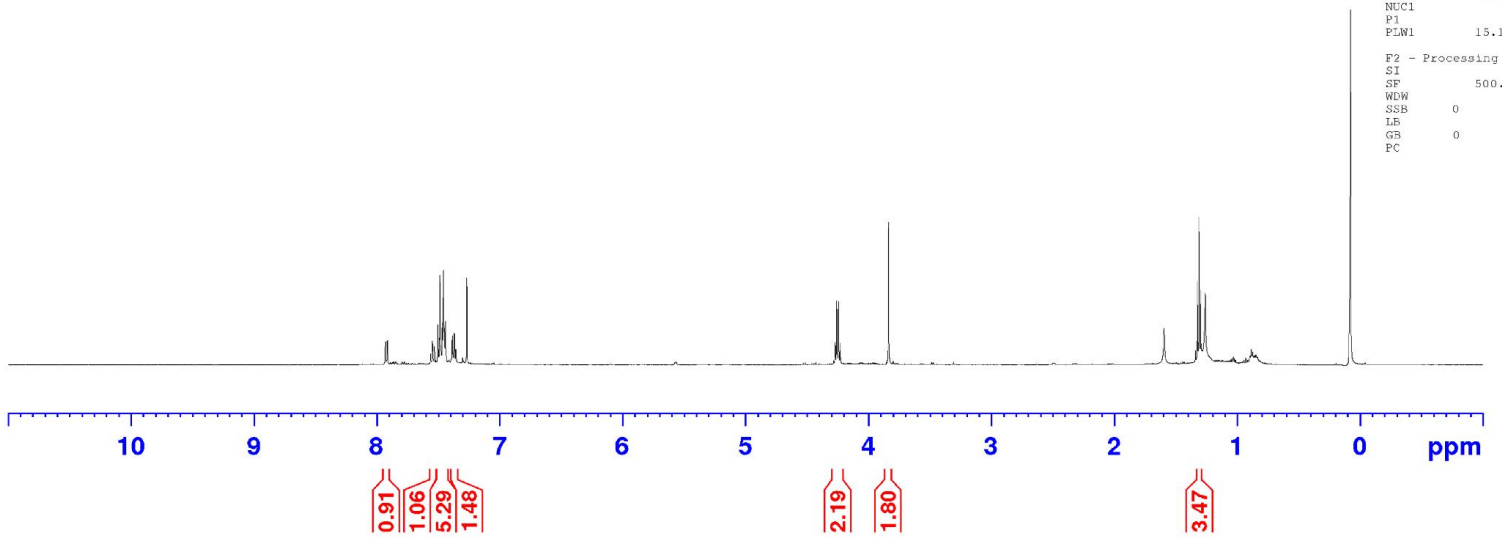

168.53
157.56
157.35
152.64
147.45
134.54
131.13
130.99
129.04
128.74
128.06
127.70
124.52
122.55
121.04
117.26
112.58
109.46
61.88
32.91
14.17
BRUKER
O
O
O
Cl
COOEt
(3e)
Current Data Parameters
NAME Nov17-2015
EXPNO 91
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151117
Time 22.21
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 293.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.8131145 MHz
NUC1 13C
P1 9.70 usec
PLW1 73.00000000 W
======== CHANNEL f2 ========
SFO2 500.3020012 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 15.13599968 W
PLW12 0.53924000 W
PLW13 0.34512001 W
F2 - Processing parameters
SI 32768
SF 125.8005351 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

3e SY-GS2-112

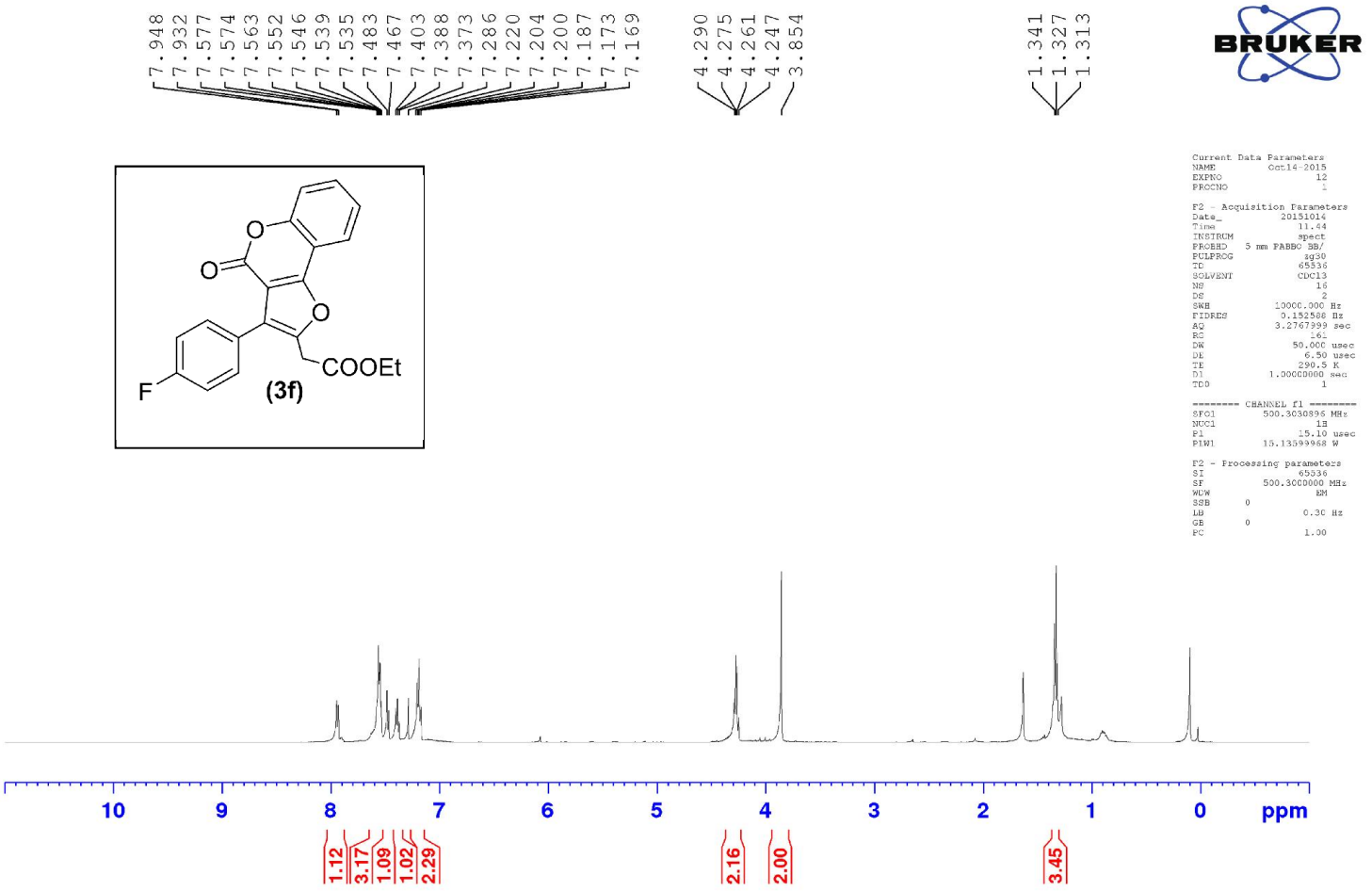


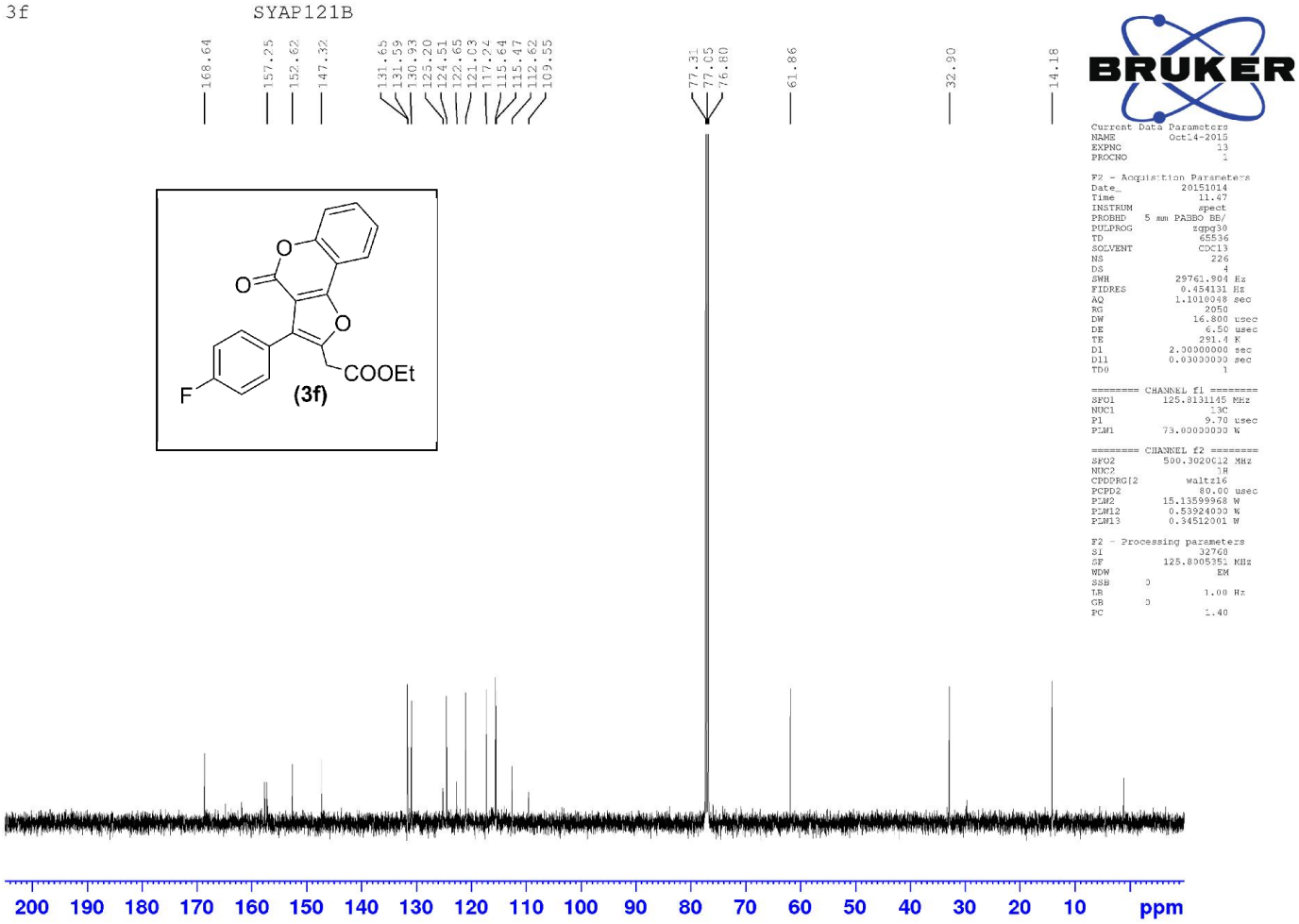



```
Current Data Parameters
NAME                  Nov17-2015
EXPNO                         90
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20151117
Time                       21.26
INSTRUM                    spect
PROBHD     5 mm PABBO BB/
PULPROG                    zg30
TD                        65536
SOLVENT                   CDCl3
NS                           16
DS                            2
SWH                  10000.000 Hz
FIDRES                0.152588 Hz
AQ                    3.2767999 sec
RG                          181
DW                       50.000 usec
DE                        6.50 usec
TE                       291.9 K
D1                    1.00000000 sec
TD0                            1

======== CHANNEL f1 ========
SFO1                500.3030896 MHz
NUC1                        1H
P1                       15.10 usec
PLW1                15.13599968 W

F2 - Processing parameters
SI                        65536
SF                  500.3000093 MHz
WDW                          EM
SSB                0
LB                        0.30 Hz
GB                 0
PC                        1.00
```

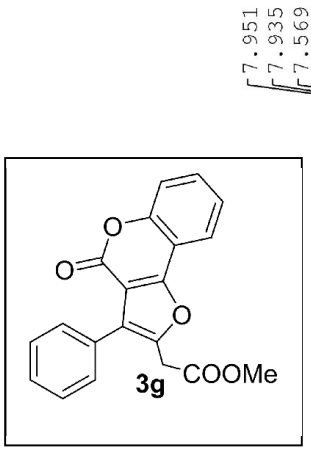


3f SYAP121B


7.948
7.932
7.577
7.574
7.563
7.552
7.546
7.539
7.535
7.483
7.467
7.403
7.388
7.373
7.286
7.220
7.204
7.200
7.187
7.173
7.169
4.290
4.275
4.261
4.247
3.854
1.341
1.327
1.313
BRUKER
O
O
O
F
COOEt
(3f)
Current Data Parameters
NAME Oct14-2015
EXPNO 12
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151014
Time 11.44
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 162
DW 50.000 usec
DE 6.50 usec
TE 290.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.3030896 MHz
NUC1 1H
P1 15.10 usec
PLW1 15.13599968 W
F2 - Processing parameters
SI 65536
SF 500.3000000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
0
ppm
1.12
3.17
1.09
1.02
2.29
2.16
2.00
3.45

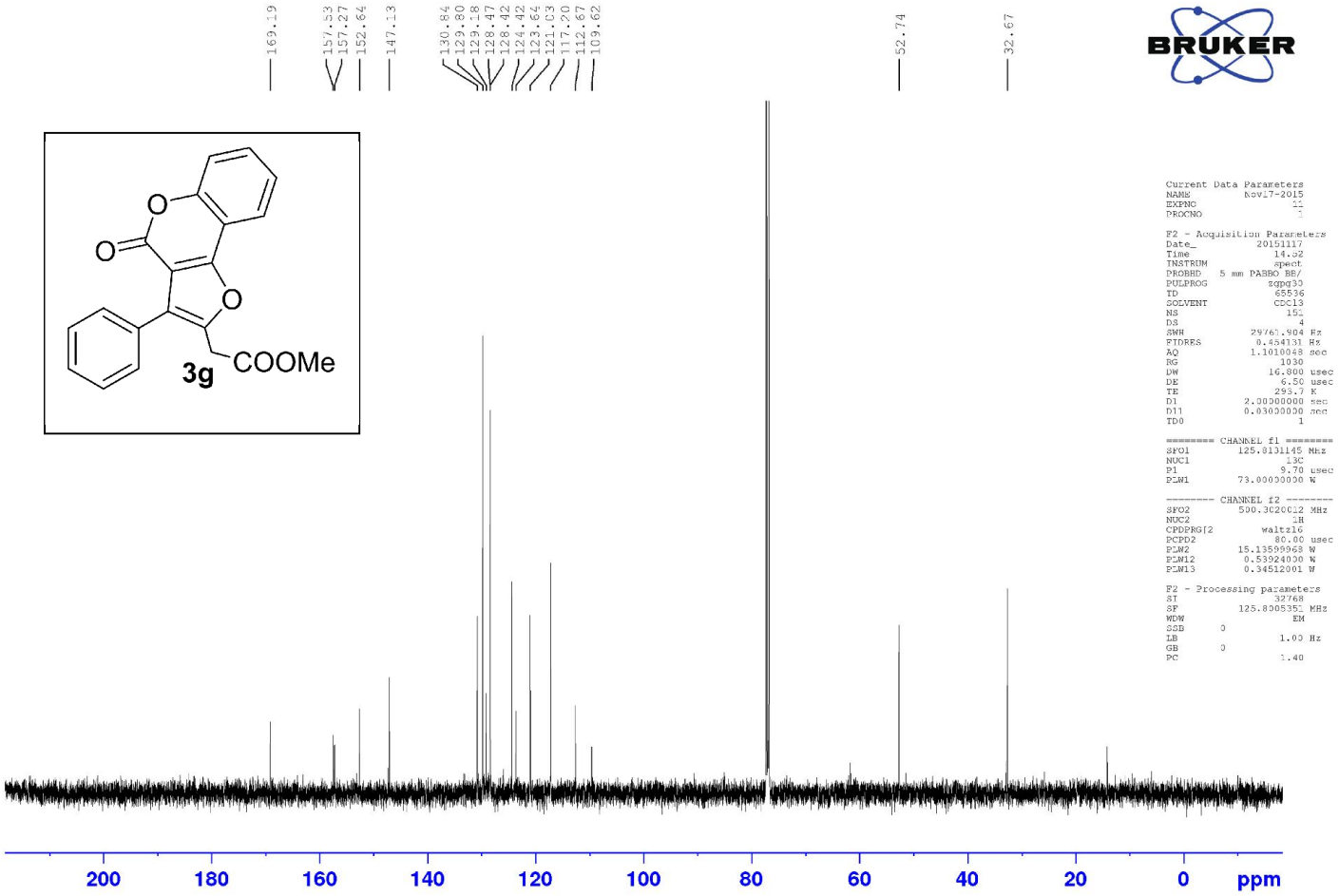

3f
SYAP121B
168.64
157.25
152.62
147.32
131.65
131.59
130.93
123.20
124.51
122.65
121.03
117.24
115.64
115.47
112.62
109.55
77.31
77.05
76.80
61.86
32.90
14.18
BRUKER
Current Data Parameters
NAME Oct14-2015
EXPNO 13
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151014
Time 11.47
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 226
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 291.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.8131145 MHz
NUC1 13C
P1 9.70 usec
PLW1 73.00000000 W
======== CHANNEL f2 ========
SFO2 500.3020012 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 15.13599968 W
PLW12 0.53924000 W
PLW13 0.34512001 W
F2 - Processing parameters
SI 32768
SF 125.8005351 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
O
O
COOEt
F
(3f)
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

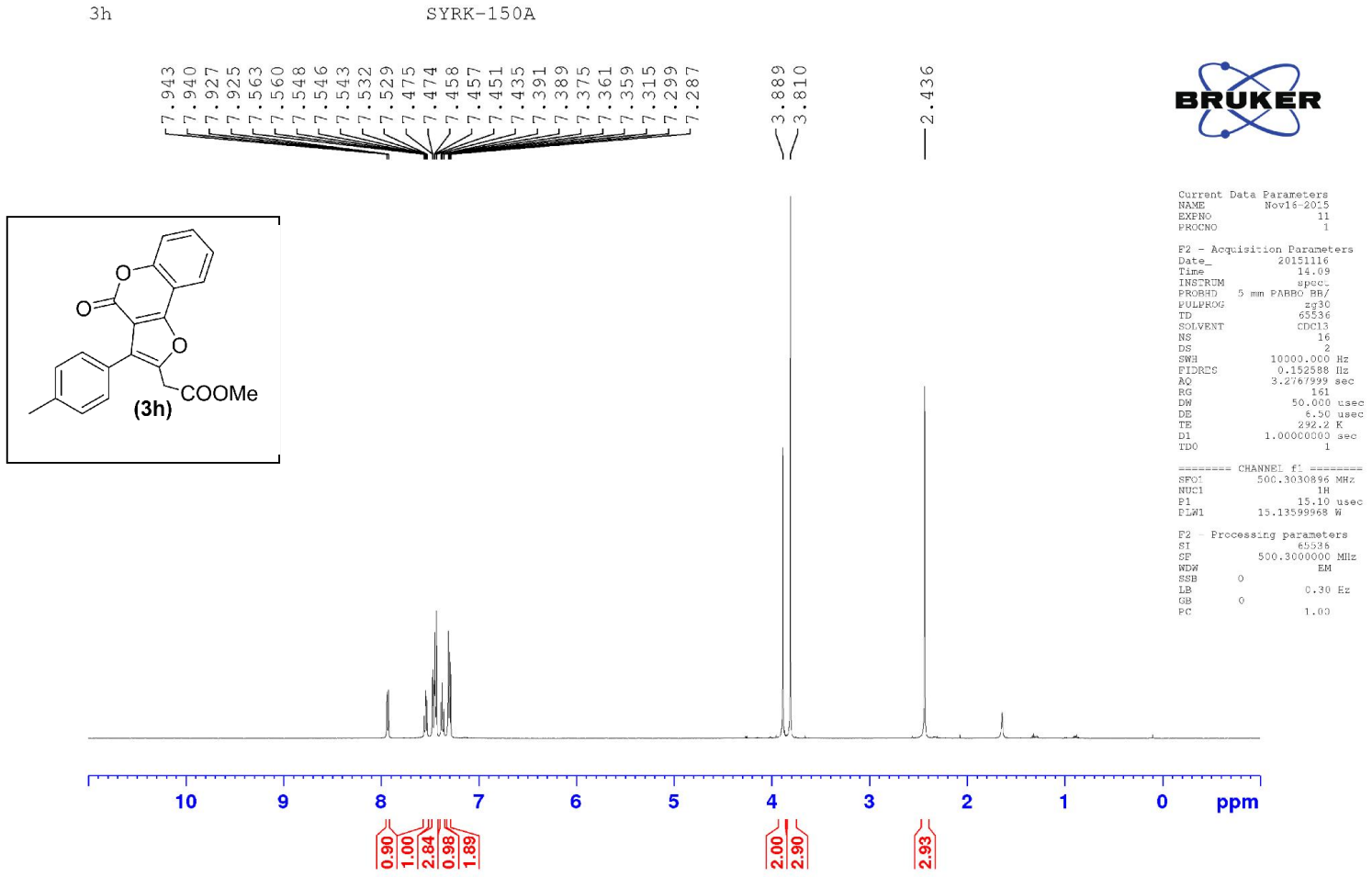
3g SYRK_152B


O
O
O
3g
COOMe

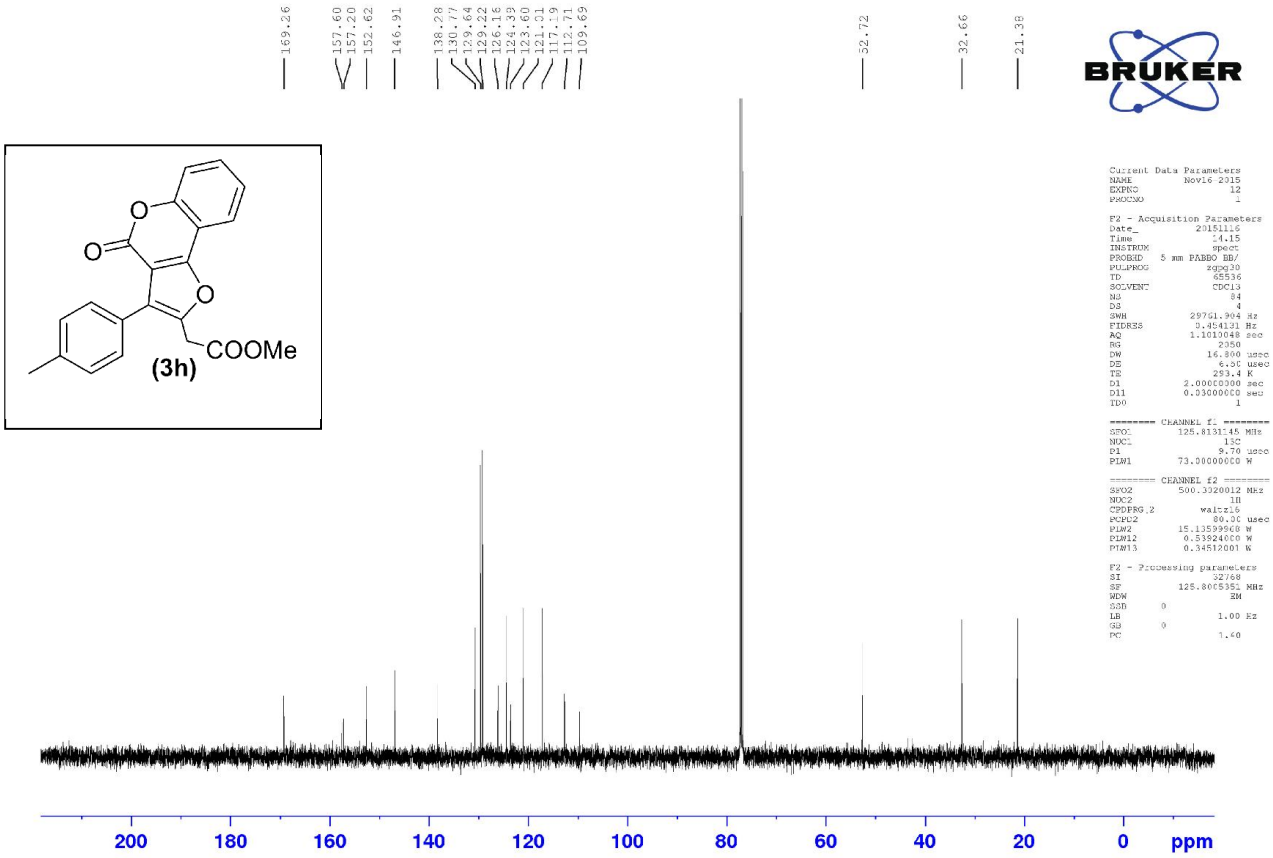
7.951
7.935
7.569
7.566
7.555
7.553
7.541
7.516
7.501
7.485
7.482
7.464
7.458
7.443
7.430
7.429
7.398
7.383
7.368
3.898
3.815
BRUKER
Current Data Parameters
NAME Nov17-2015
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151117
Time 14.42
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 161
DW 50.000 usec
DE 6.50 usec
TE 292.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.3030896 MHz
NUC1 1H
P1 15.10 usec
PLW1 15.13599968 W
F2 - Processing parameters
SI 65536
SF 500.3000000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
0
ppm
1.13
1.92
3.05
1.92
1.13
2.00
3.00

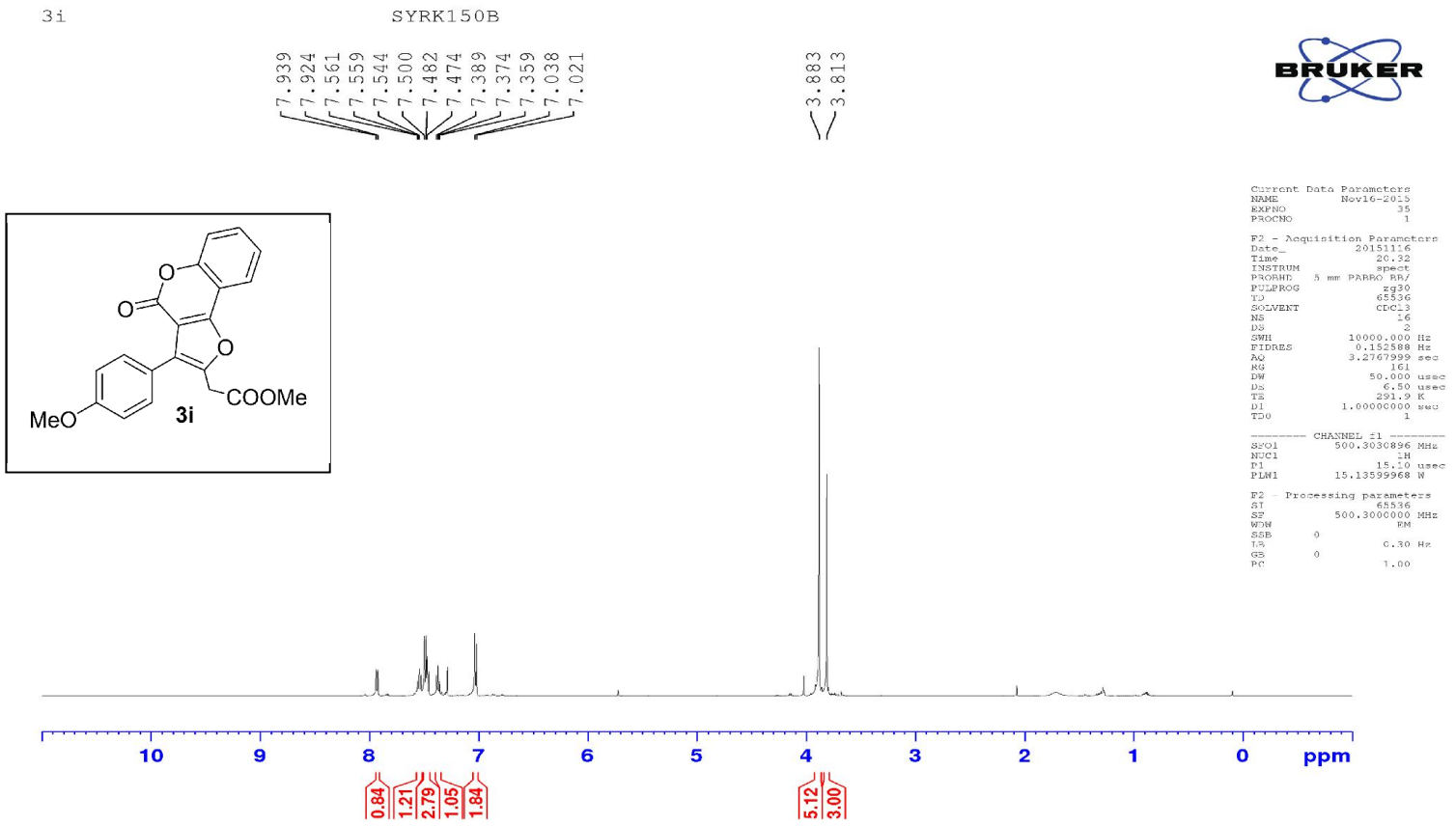
SYRK_152B

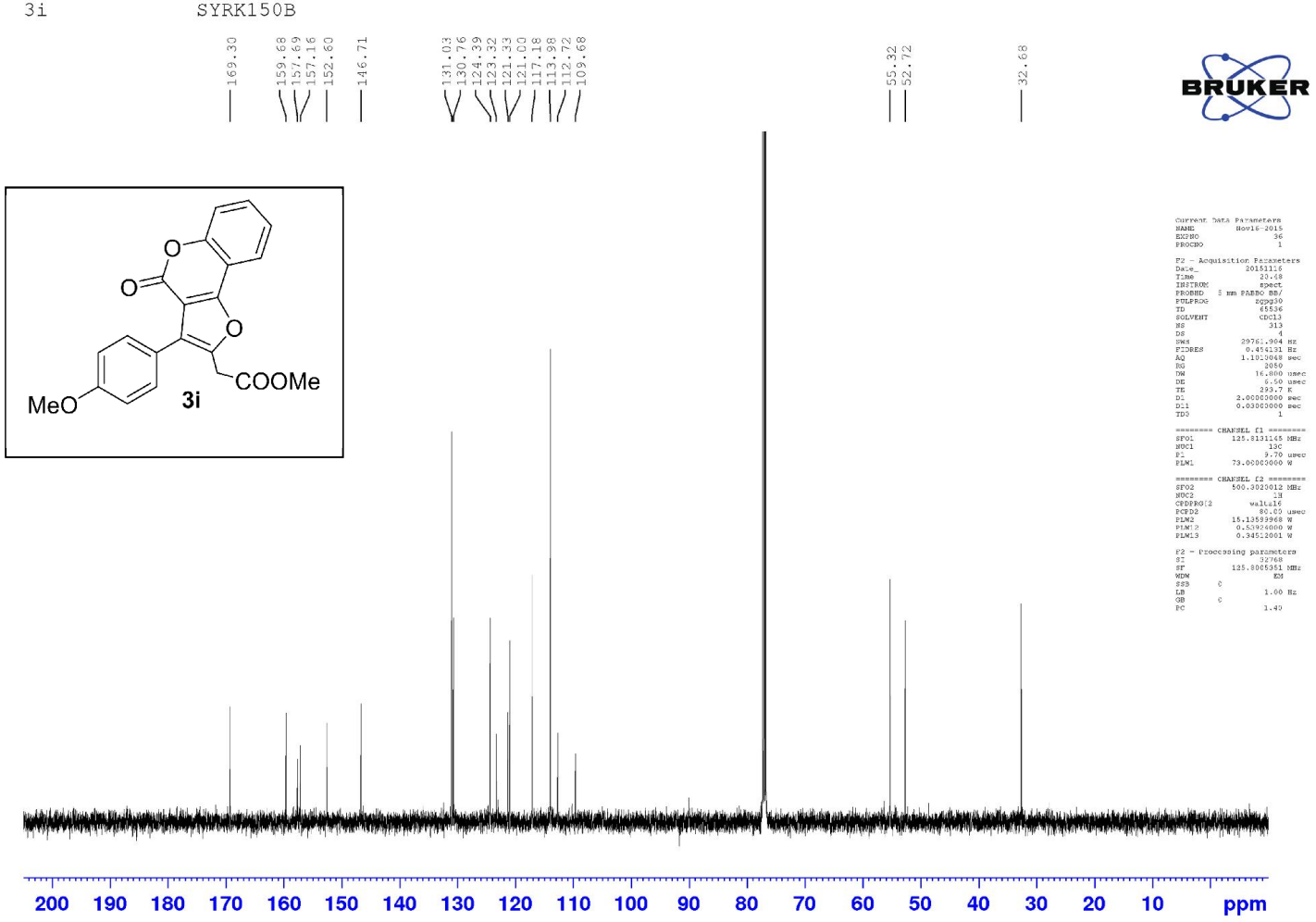

169.19
157.53
157.27
152.64
147.13
130.84
129.80
129.18
128.47
128.42
124.42
123.64
121.03
117.20
112.67
109.62
52.74
32.67
BRUKER
O
O
O
3g
COOMe
Current Data Parameters
NAME kov17-2015
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151117
Time 14.52
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 152
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 1030
DW 16.800 usec
DE 6.50 usec
TE 293.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.8131145 MHz
NUC1 13C
P1 9.70 usec
PLW1 73.00000000 W
======== CHANNEL f2 ========
SFO2 500.3020012 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 15.13599968 W
PLW12 0.53924000 W
PLW13 0.34512001 W
F2 - Processing parameters
SI 32768
SF 125.8005351 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

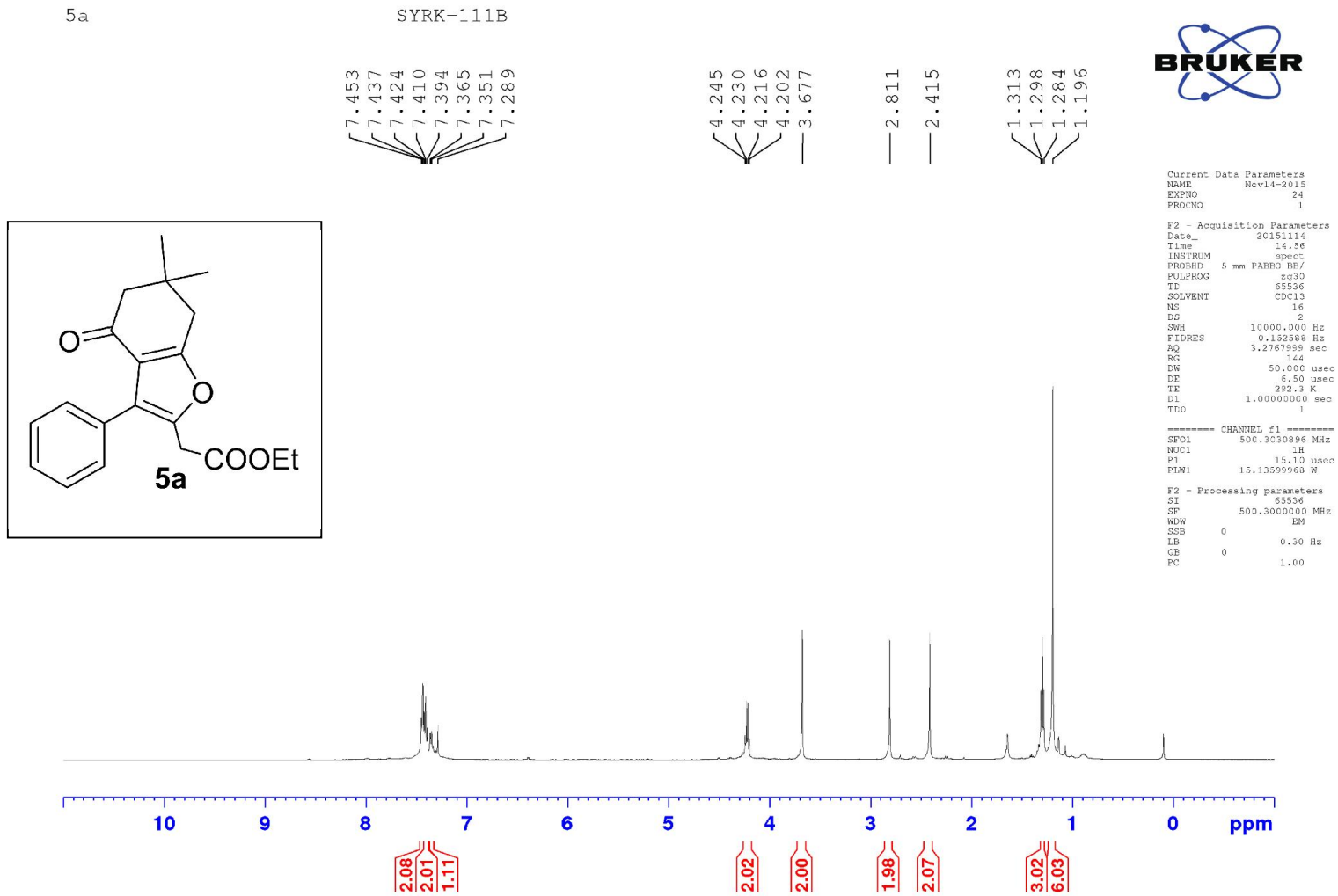

3h
SYRK-150A
7.943
7.940
7.927
7.925
7.563
7.560
7.548
7.546
7.543
7.532
7.529
7.475
7.474
7.458
7.457
7.451
7.435
7.391
7.389
7.375
7.361
7.359
7.315
7.299
7.287
3.889
3.810
2.436
BRUKER
O
O
O
COOMe
(3h)
Current Data Parameters
NAME Nov16-2015
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151116
Time 14.09
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 161
DW 50.000 usec
DE 6.50 usec
TE 292.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.3030896 MHz
NUC1 1H
P1 15.10 usec
PLW1 15.13599968 W
F2 - Processing parameters
SI 65536
SF 500.3000000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
0
ppm
0.90
1.00
2.84
0.98
1.89
2.00
2.90
2.93

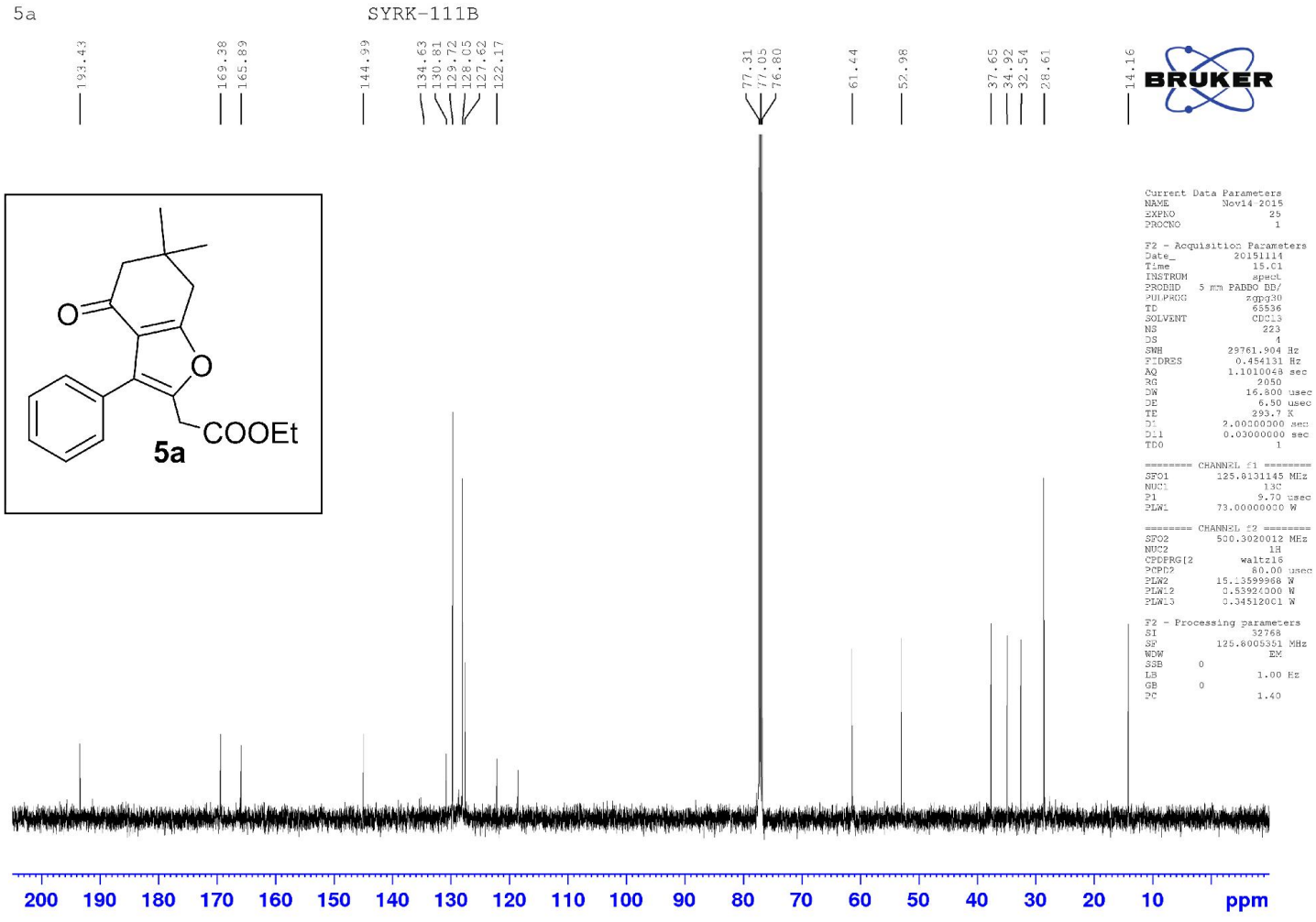
SYRK-150A

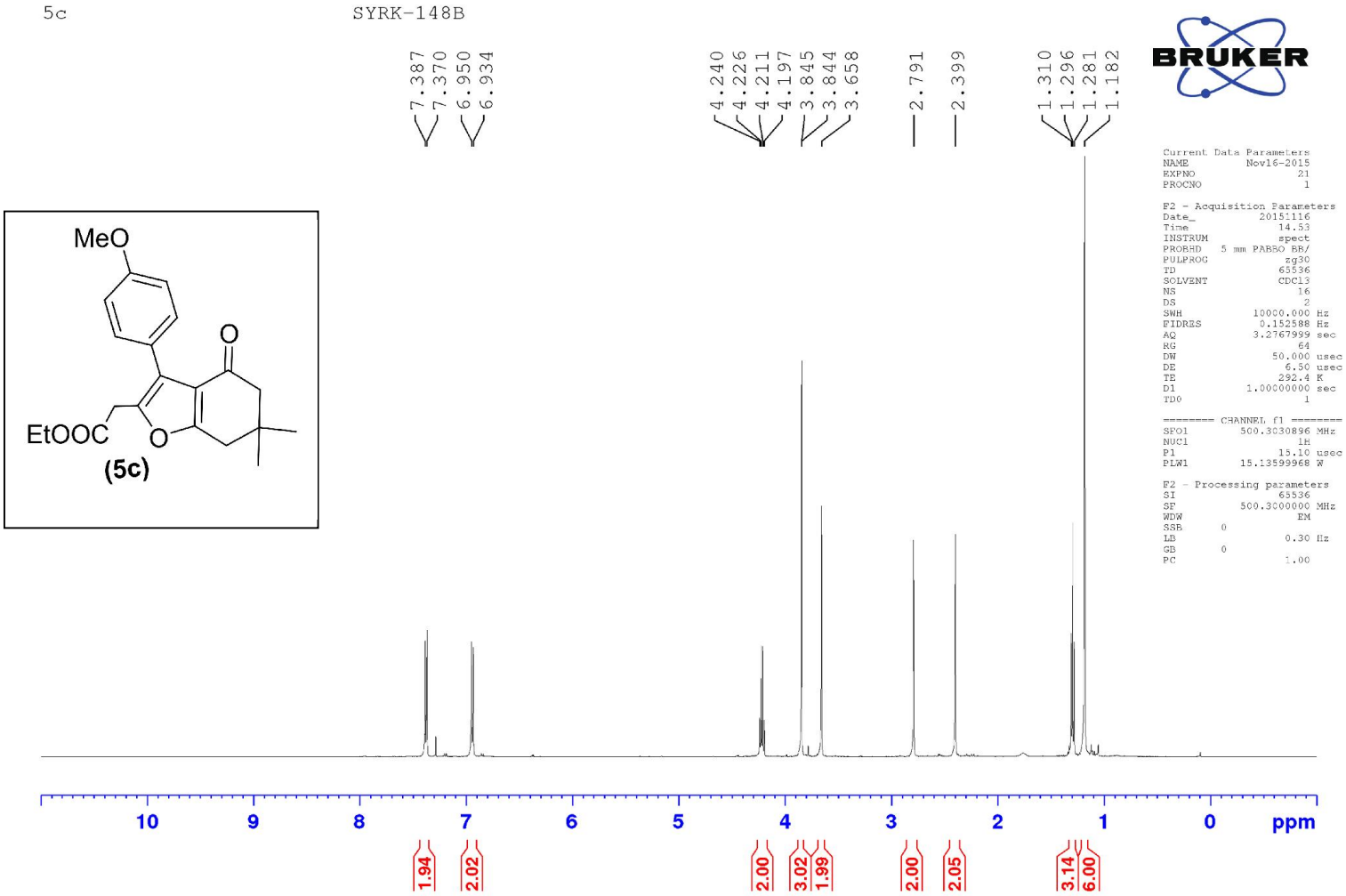

169.26
157.60
157.20
152.62
146.91
138.28
130.77
129.64
129.22
126.16
124.39
123.60
121.01
117.19
112.71
109.69
52.72
32.66
21.38
BRUKER
O
O
O
COOMe
(3h)
Current Data Parameters
NAME Nov16_2015
EXPNO 12
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151116
Time 14.15
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 84
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 293.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.8131145 MHz
NUC1 13C
P1 9.70 usec
PLW1 73.00000000 W
======== CHANNEL f2 ========
SFO2 500.3320012 MHz
NUC2 1H
CPDPRG_2 waltz16
PCPD2 80.00 usec
PLW2 15.13599968 W
PLW12 0.53924000 W
PLW13 0.34512001 W
F2 - Processing parameters
SI 32768
SF 125.8005351 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

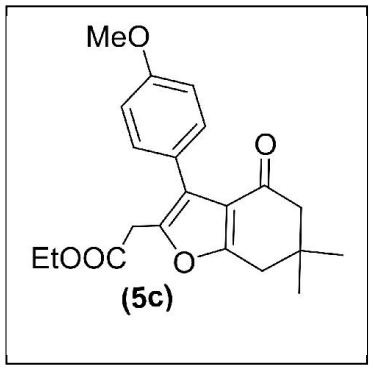

3i
SYRK150B
7.939
7.924
7.561
7.559
7.544
7.500
7.482
7.474
7.389
7.374
7.359
7.038
7.021
3.883
3.813
BRUKER
Current Data Parameters
NAME Nov16-2015
EXPNO 35
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151116
Time 20.32
INSTRUM spect
PROBHD 5 mm PABBO BA/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 161
DW 50.000 usec
DE 6.50 usec
TE 291.9 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
SFO1 500.3030896 MHz
NUC1 1H
P1 15.10 usec
PLW1 15.13599968 W
F2 - Processing parameters
SI 65536
SF 500.3000000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
O
O
MeO
COOMe
3i
10
9
8
7
6
5
4
3
2
1
0
ppm
0.84
1.21
2.79
1.05
1.84
5.12
3.00


3i
SYRK150B
169.30
159.68
157.69
157.16
152.60
146.71
131.03
130.76
124.39
123.32
121.33
121.00
117.18
113.98
112.72
109.68
55.32
52.72
32.68
BRUKER
O
O
O
MeO
COOMe
3i
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


5a
SYRK-111B
7.453
7.437
7.424
7.410
7.394
7.365
7.351
7.289
4.245
4.230
4.216
4.202
3.677
2.811
2.415
1.313
1.298
1.284
1.196
BRUKER
Current Data Parameters
NAME Nov14-2015
EXPNO 24
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151114
Time 14.56
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 144
DW 50.000 usec
DE 6.50 usec
TE 292.3 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
SFO1 500.3030896 MHz
NUC1 1H
P1 15.10 usec
PLW1 15.13599968 W
F2 - Processing parameters
SI 65536
SF 500.3000000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
O
COOEt
5a
10
9
8
7
6
5
4
3
2
1
0
ppm
2.08
2.01
1.11
2.02
2.00
1.98
2.07
3.02
6.03


5a
SYRK-111B
193.43
169.38
165.89
144.99
134.63
130.81
129.72
128.05
127.62
122.17
77.31
77.05
76.80
61.44
52.98
37.65
34.92
32.54
28.61
14.16
BRUKER
O
O
COOEt
5a
Current Data Parameters
NAME Nov14-2015
EXPNO 25
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151114
Time 15.01
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 223
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 293.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.0131145 MHz
NUC1 13C
P1 9.70 usec
PLW1 73.00000000 W
======== CHANNEL f2 ========
SFO2 500.3020012 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 15.13599968 W
PLW12 0.53920300 W
PLW13 0.34512001 W
F2 - Processing parameters
SI 32768
SF 125.8005351 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


5c
SYRK-148B
7.387
7.370
6.950
6.934
4.240
4.226
4.211
4.197
3.845
3.844
3.658
2.791
2.399
1.310
1.296
1.281
1.182
BRUKER
MeO
O
EtOOC
O
(5c)
Current Data Parameters
NAME Nov16-2015
EXPNO 21
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151116
Time 14.53
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 64
DW 50.000 usec
DE 6.50 usec
TE 292.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.3030896 MHz
NUC1 1H
P1 15.10 usec
PLW1 15.13599968 W
F2 - Processing parameters
SI 65536
SF 500.3000000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
0
ppm
1.94
2.02
2.00
3.02
1.99
2.00
2.05
3.14
6.00

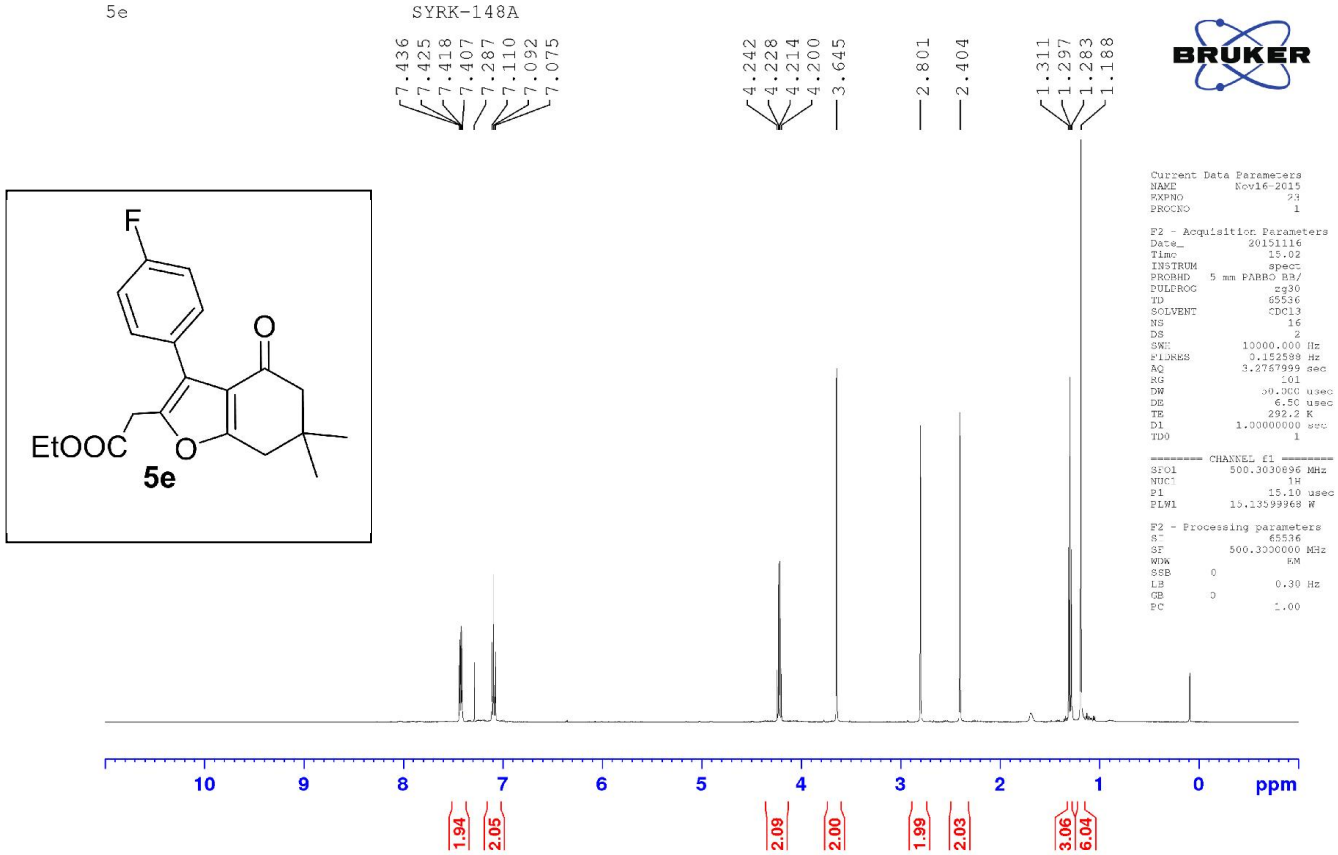


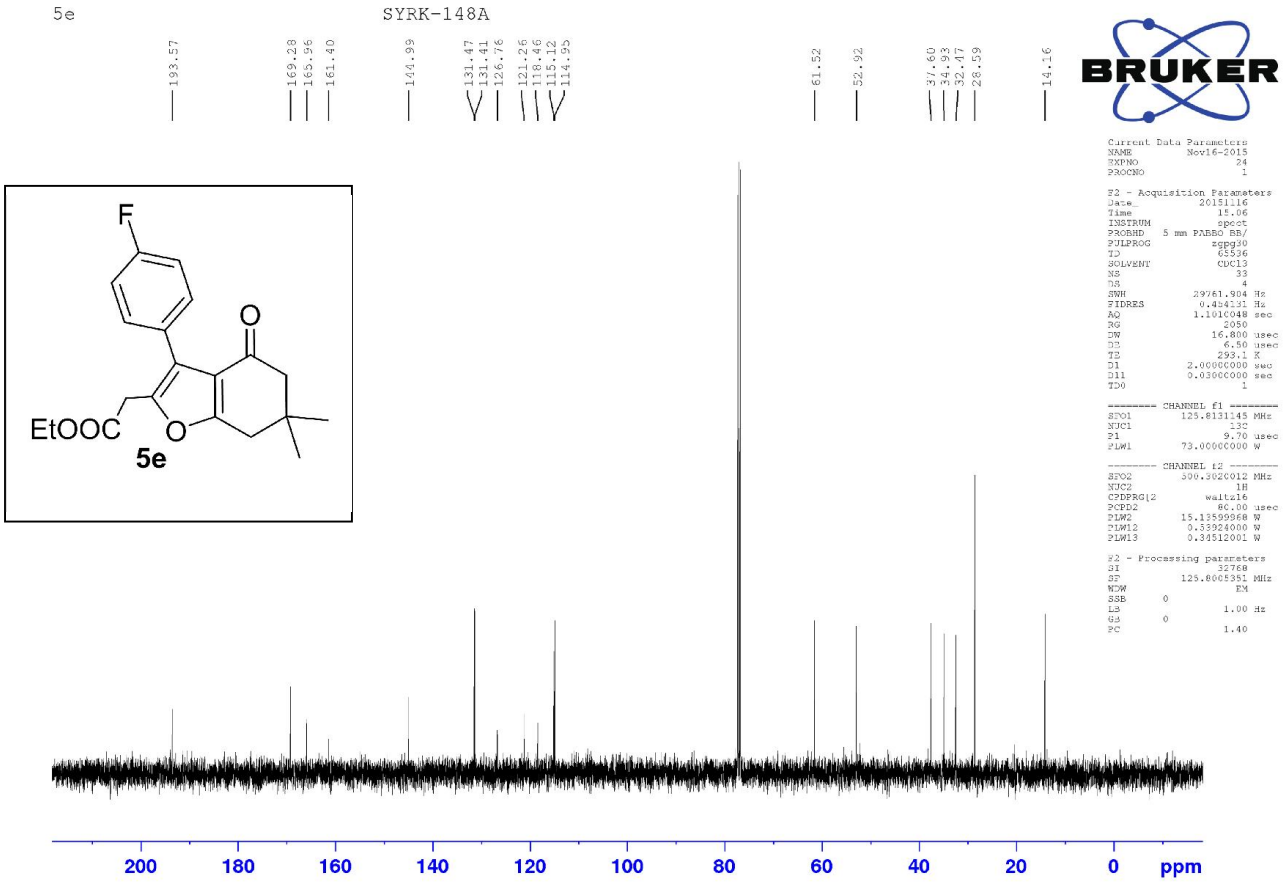
5C SYRK-148B

193.56 169.49 165.82 159.08 144.57 130.90 123.04 121.78 118.57 113.53 61.42 55.26 52.98 37.65 34.88 32.54 28.60 14.17

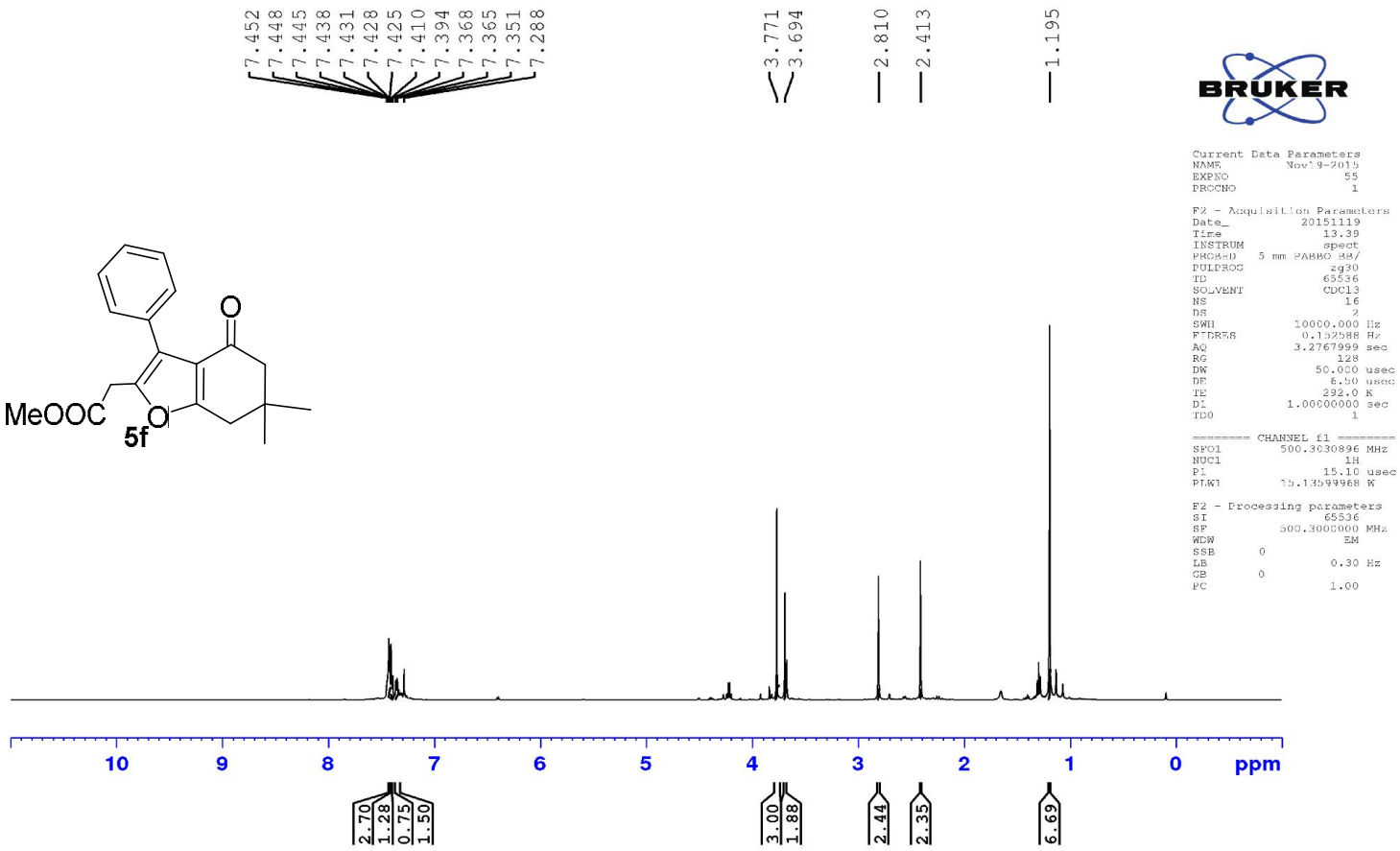
BRUKER


```
Current Data Parameters
NAME                  Nov16-2015
EXPNO                         22
PROCNO                         1

F2 - Acquisition Parameters
Date_               20151116
Time                      14.56
INSTRUM                   spect
PROBHD    5 mm PABBO BB/
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                         24
DS                          4
SWH                 29761.904 Hz
FIDRES                0.454131 Hz
AQ                    1.1010048 sec
RG                         2050
DW                       16.800 usec
DE                        6.50 usec
TE                       293.0 K
CL                    2.00000000 sec
CL1                   0.03000000 sec
TD0                            1

======== CHANNEL f1 ========
SFO1                125.8131145 MHz
NUC1                       13C
PL1                       9.70 usec
PLW1                73.00000000 W

======== CHANNEL f2 ========
SFO2                500.3020012 MHz
NUC2                       1H
CPDPRG[2                waltz16
PCPD2                    80.00 usec
PLW2                15.13599368 W
PLW12                0.53924000 W
PLW13                0.34512001 W

F2 - Processing parameters
SI                         32768
SF                  125.8005351 MHz
WDW                           EM
SSB                0
LB                        1.00 Hz
GB                 0
PC                        1.40
```

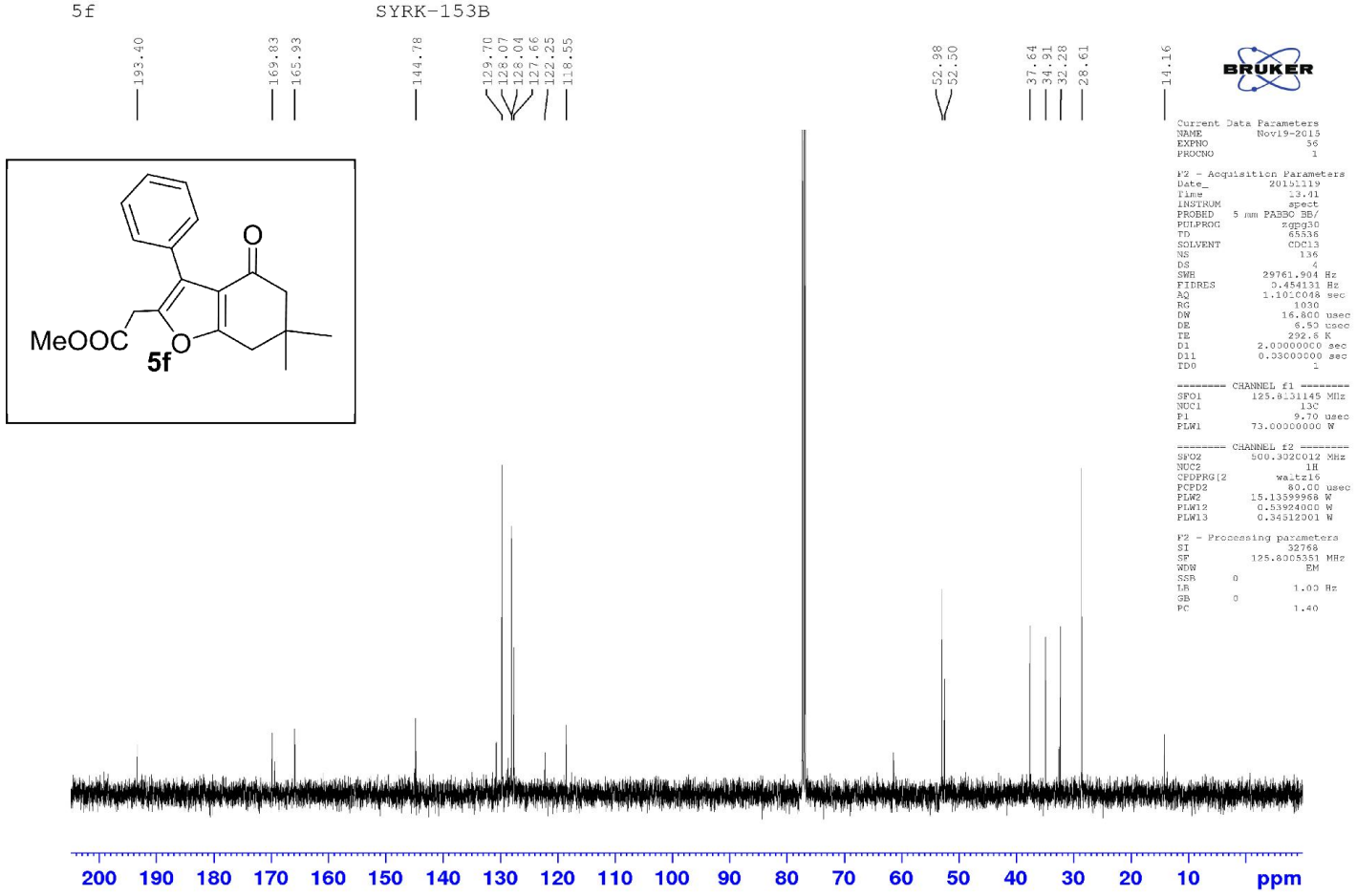

5e
SYRK-148A
7.436
7.425
7.418
7.407
7.287
7.110
7.092
7.075
4.242
4.228
4.214
4.200
3.645
2.801
2.404
1.311
1.297
1.283
1.188
BRUKER
F
O
EtOOC
O
5e
Current Data Parameters
NAME Nov16-2015
EXPNO 23
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151116
Time 15.02
INSTRUM spect
PROBHD 5 mm PABBO BA/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 101
DW 50.000 usec
DE 6.50 usec
TE 292.2 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
SFO1 500.3030896 MHz
NUC1 1H
P1 15.10 usec
PLW1 15.13599968 W
F2 - Processing parameters
SI 65536
SF 500.3000000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
0
ppm
1.94
2.05
2.09
2.00
1.99
2.03
3.06
6.04


5e
SYRK-148A
193.57
169.28
165.96
161.40
144.99
131.47
131.41
126.76
121.26
118.46
115.12
114.95
61.52
52.92
37.60
34.93
32.47
28.59
14.16
BRUKER
F
O
EtOOC
O
5e
Current Data Parameters
NAME Nov16-2015
EXPNO 24
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151116
Time 15.06
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 33
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 293.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.8131145 MHz
NUC1 13C
P1 9.70 usec
PLW1 73.00000000 W
======== CHANNEL f2 ========
SFO2 500.3020012 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 15.13599968 W
PLW12 0.33924000 W
PLW13 0.30512001 W
F2 - Processing parameters
SI 32768
SF 125.8005351 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

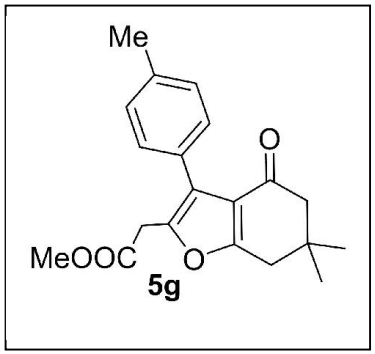
SYRK-153B



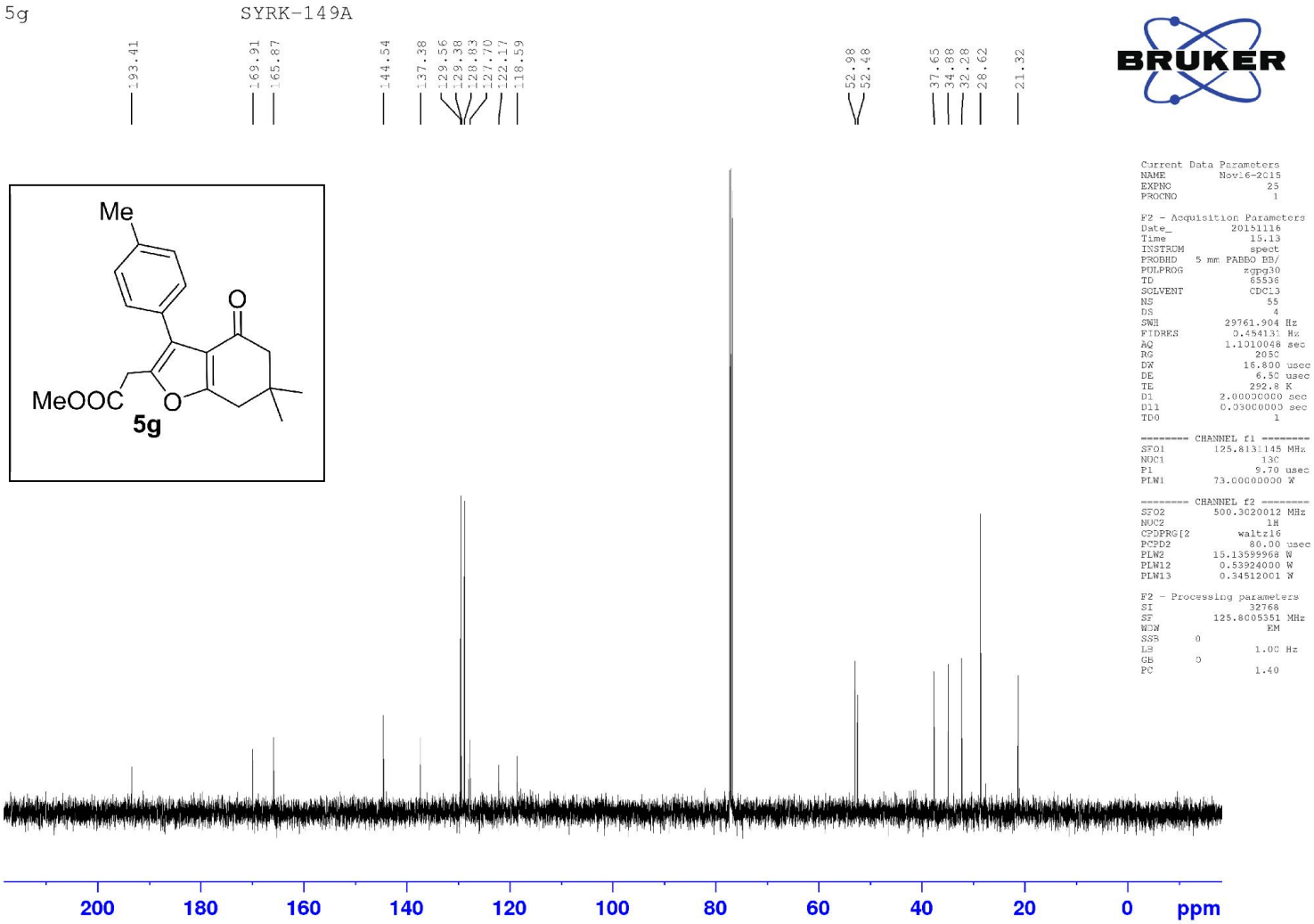

```
Current Data Parameters
NAME                  Nov19-2015
EXPNO                         55
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20151119
Time                       13.39
INSTRUM                    spect
PROBHD     5 mm PABBO BB/
PULPROG                    zg30
TD                        65536
SOLVENT                   CDCl3
NS                           16
DS                            2
SWH                   10000.000 Hz
FIDRES                0.152588 Hz
AQ                    3.2767999 sec
RG                          128
DW                     50.000 usec
DE                      6.50 usec
TE                      292.0 K
D1                  1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1                500.3030896 MHz
NUC1                        1H
P1                       15.10 usec
PLW1                15.13599968 W

F2 - Processing parameters
SI                        65536
SF                  500.3000000 MHz
WDW                          EM
SSB                 0
LB                        0.30 Hz
GB                  0
PC                        1.00
```

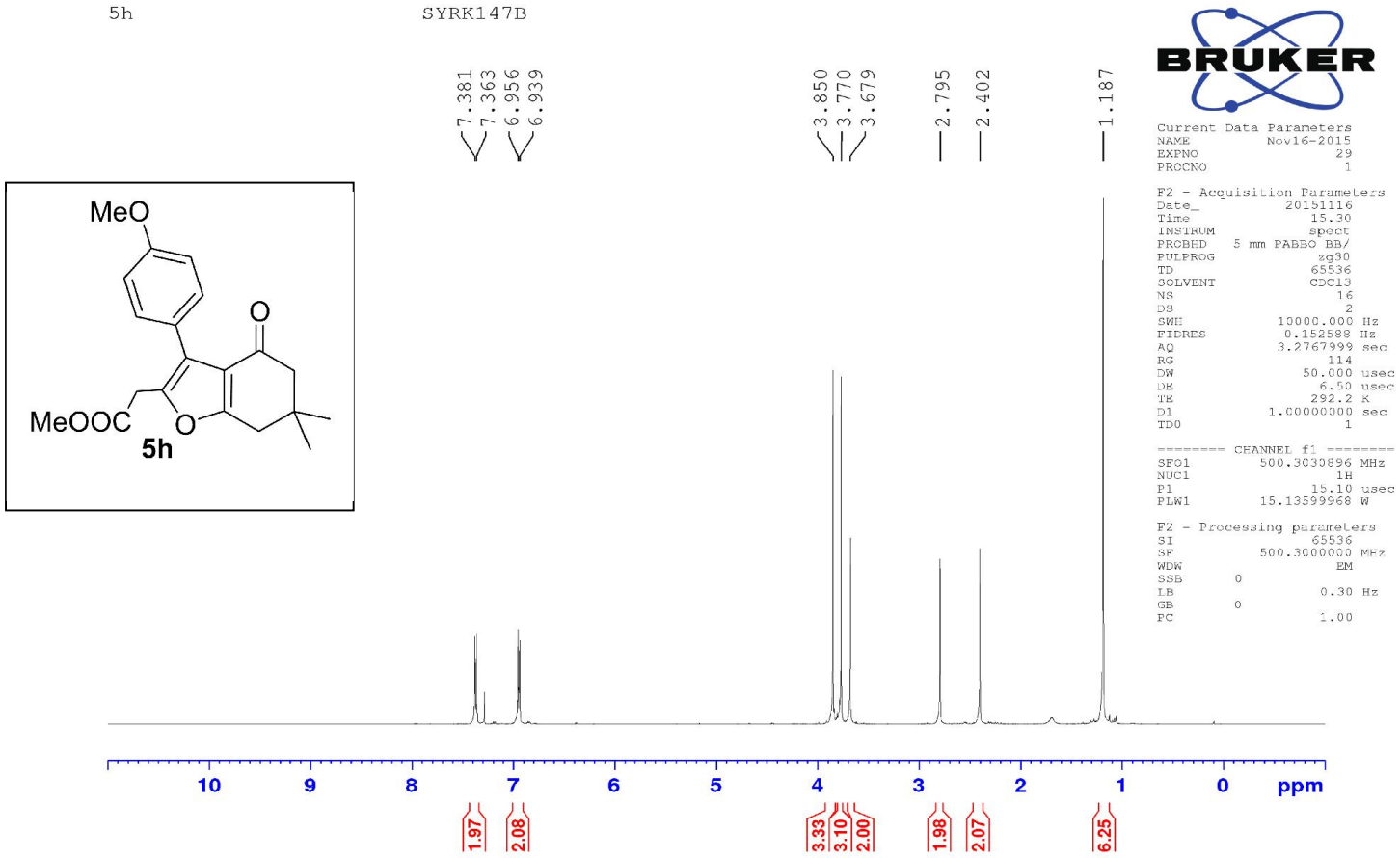

5f
SYRK-153B
193.40
169.83
165.93
144.78
129.70
128.07
128.04
127.66
122.25
118.55
52.98
52.50
37.64
34.91
32.28
28.61
14.16
BRUKER
MeOOC
O
O
5f
Current Data Parameters
NAME Nov19-2015
EXPNO 56
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151119
Time 13.41
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 136
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1012048 sec
RG 1030
DW 16.800 usec
DE 6.50 usec
TE 292.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
SFO1 125.8131145 MHz
NUC1 13C
P1 9.70 usec
PLW1 73.00000000 W
CHANNEL f2
SFO2 500.3020012 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 15.13599968 W
PLW12 0.53924000 W
PLW13 0.34512001 W
F2 - Processing parameters
SI 32768
SF 125.8005351 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

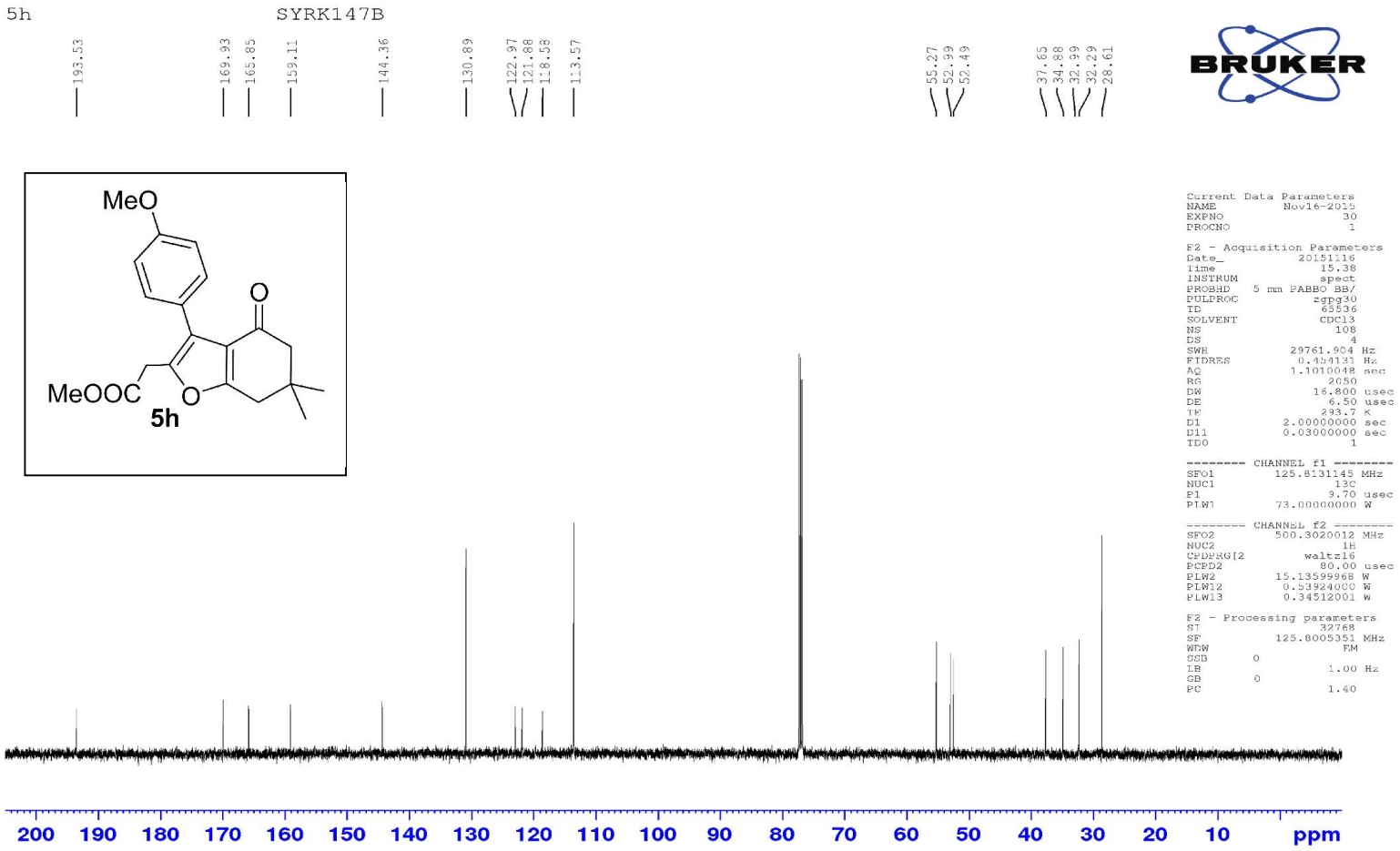
5g SYRK-149A

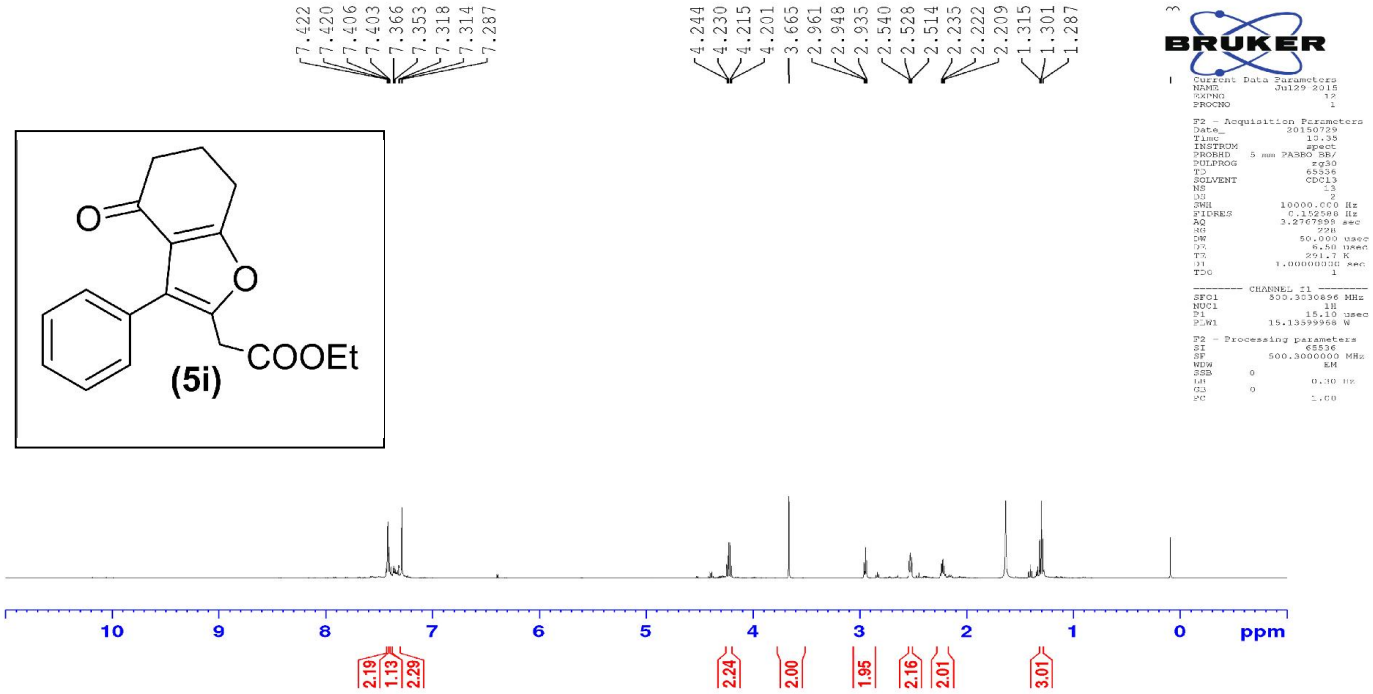

Me
O
MeOOC
O
5g

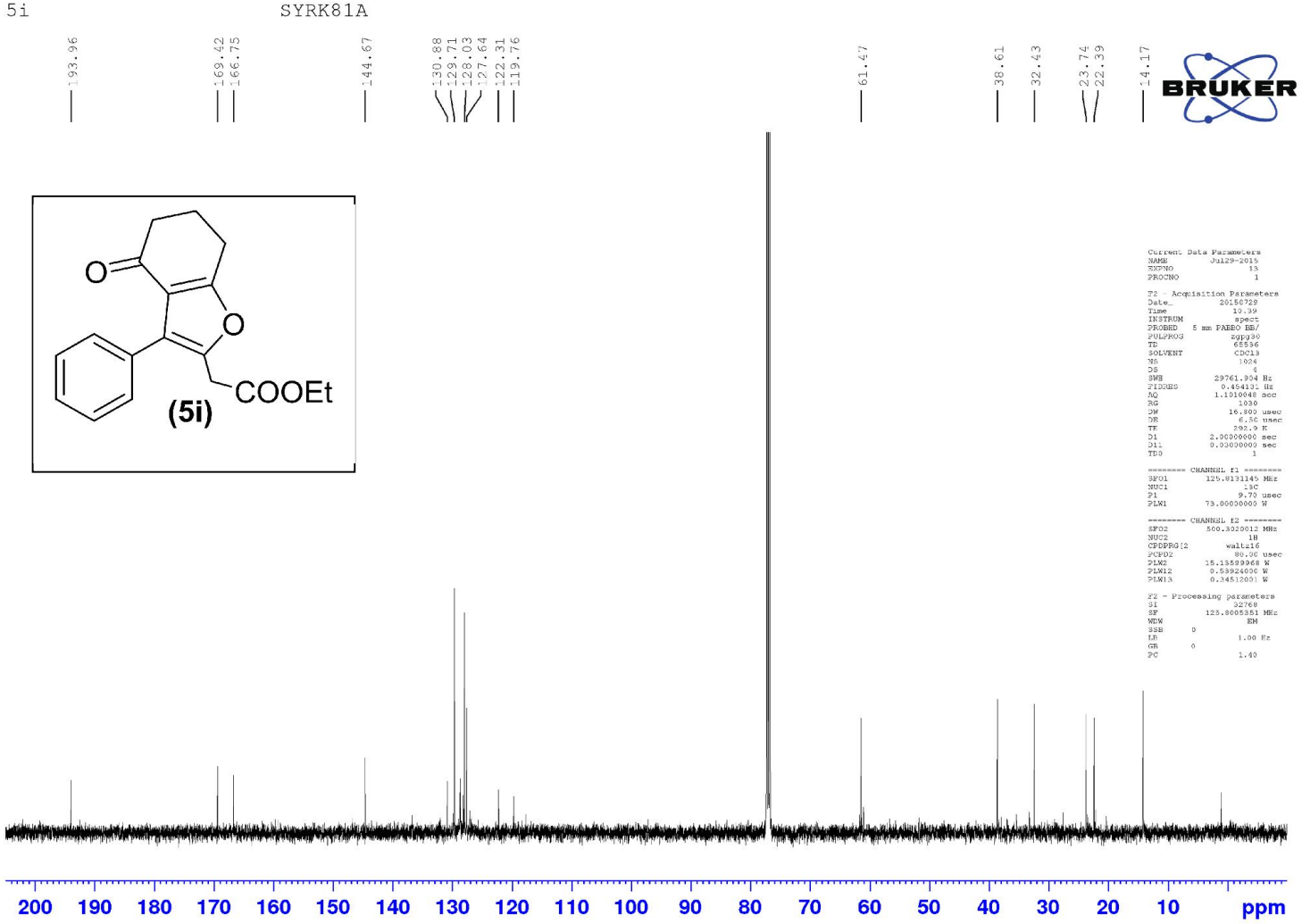
7.334
7.318
7.288
7.228
7.212
3.768
3.686
2.801
2.406
2.395
1.191
BRUKER
Current Data Parameters
NAME Nov16-2015
EXPNO 26
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151116
Time 15.18
INSTRUM spect
PROBHD 5 mm DABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 114
DW 50.000 usec
DE 6.50 usec
TE 292.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.3030896 MHz
NUC1 1H
P1 15.10 usec
PLW1 15.13599968 W
F2 - Processing parameters
SI 65536
SF 500.3000000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
0
ppm
1.96
2.02
2.82
2.01
1.96
1.96
2.88
6.00


5g
SYRK-149A
193.41
169.91
165.87
144.54
137.38
129.56
129.38
128.83
127.70
122.17
118.59
52.98
52.48
37.65
34.88
32.28
28.62
21.32
BRUKER
Me
O
MeOOC
O
5g
Current Data Parameters
NAME Nov16-2015
EXPNO 25
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151116
Time 15.13
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 55
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 292.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
SFO1 125.8131145 MHz
NUC1 13C
P1 9.70 usec
PLW1 73.00000000 W
CHANNEL f2
SFO2 500.3020012 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 15.13599968 W
PLW12 0.53924000 W
PLW13 0.34512001 W
F2 - Processing parameters
SI 32768
SF 125.8005351 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

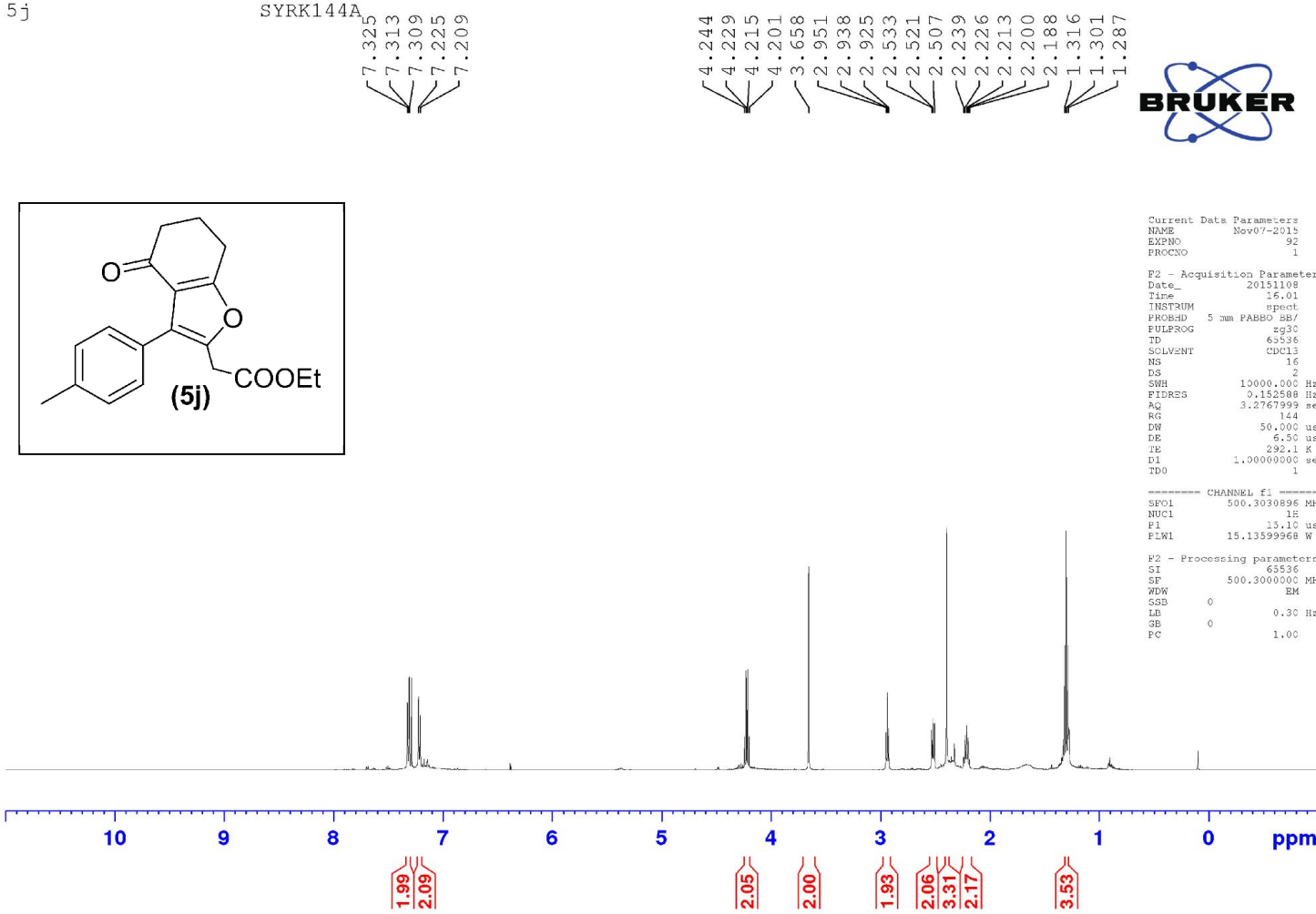

5h
SYRK147B
7.381
7.363
6.956
6.939
3.850
3.770
3.679
2.795
2.402
1.187
MeO
O
MeOOC
O
5h
BRUKER
Current Data Parameters
NAME Nov16-2015
EXPNO 29
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151116
Time 15.30
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 114
DW 50.000 usec
DE 6.50 usec
TE 292.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.3030896 MHz
NUC1 1H
P1 15.10 usec
PLW1 15.13599968 W
F2 - Processing parameters
SI 65536
SF 500.3000000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
0
ppm
1.97
2.08
3.33
3.10
2.00
1.98
2.07
6.25


5h
SYRK147B
193.53
169.93
165.85
159.11
144.36
130.89
122.97
121.88
118.58
113.57
55.27
52.99
52.49
37.65
34.88
32.99
32.29
28.61
BRUKER
MeO
O
MeOOC
O
5h
Current Data Parameters
NAME Nov16-2015
EXPNO 30
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151116
Time 15.38
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 108
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 293.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
SFO1 125.8131145 MHz
NUC1 13C
P1 9.70 usec
PLW1 73.00000000 W
CHANNEL f2
SFO2 500.3020012 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 15.13599968 W
PLW12 0.53924000 W
PLW13 0.34512001 W
F2 - Processing parameters
SI 32768
SF 125.8005351 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

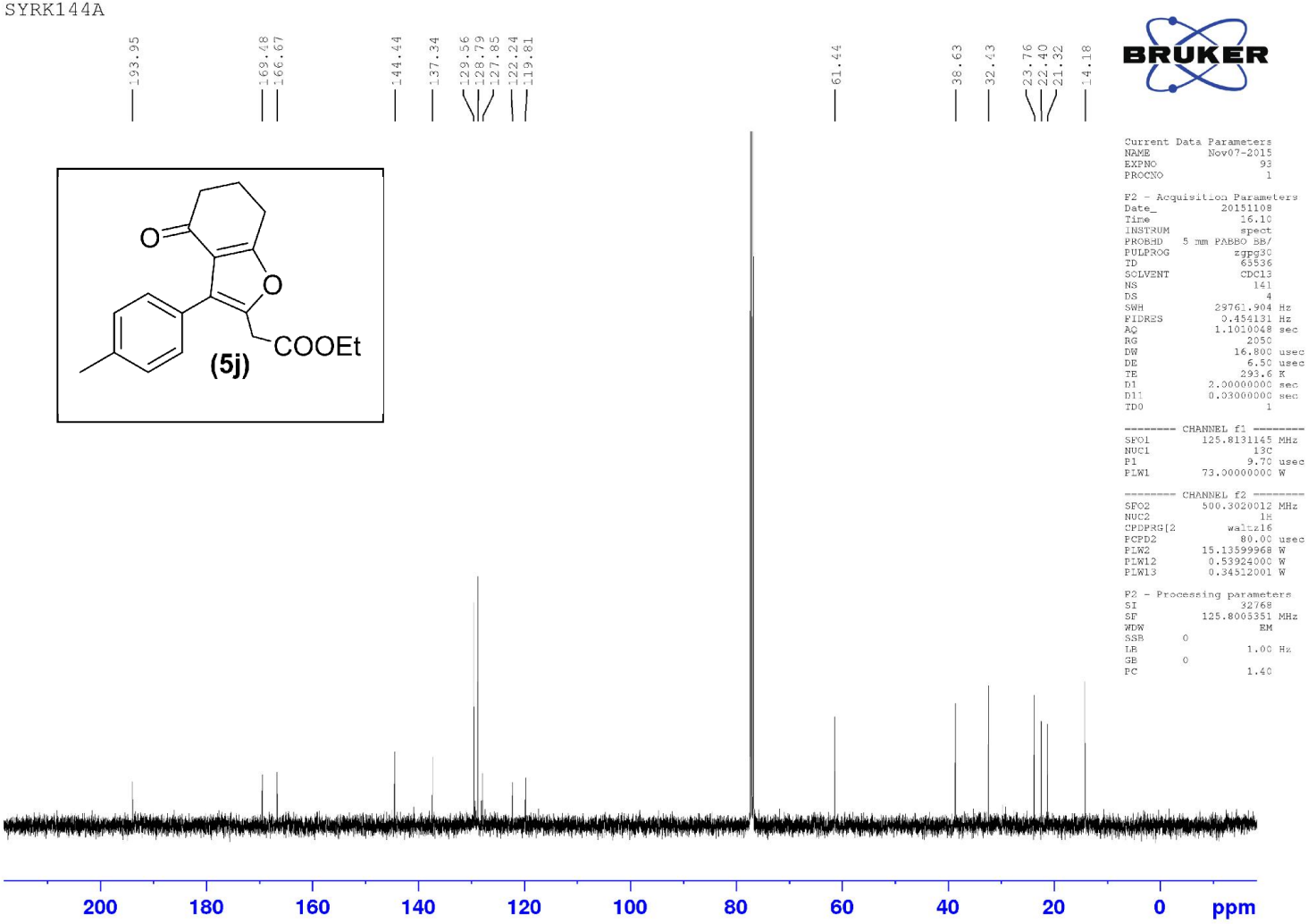

5i
SYRK81A
7.422
7.420
7.406
7.403
7.366
7.353
7.318
7.314
7.287
4.244
4.230
4.215
4.201
3.665
2.961
2.948
2.935
2.540
2.528
2.514
2.235
2.222
2.209
1.315
1.301
1.287
BRUKER
Current Data Parameters
NAME Jul29 2015
EXPNO 12
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150729
Time 13.35
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 2
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 2048
DW 50.000 usec
DE 6.50 usec
TE 301.7 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
SFO1 500.3030896 MHz
NUC1 1H
P1 15.10 usec
PLW1 15.13599968 W
F2 - Processing parameters
SI 65536
SF 500.3000000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
O
COOEt
(5i)
10
9
8
7
6
5
4
3
2
1
0
ppm
2.19
1.13
2.29
2.24
2.00
1.95
2.16
2.01
3.01

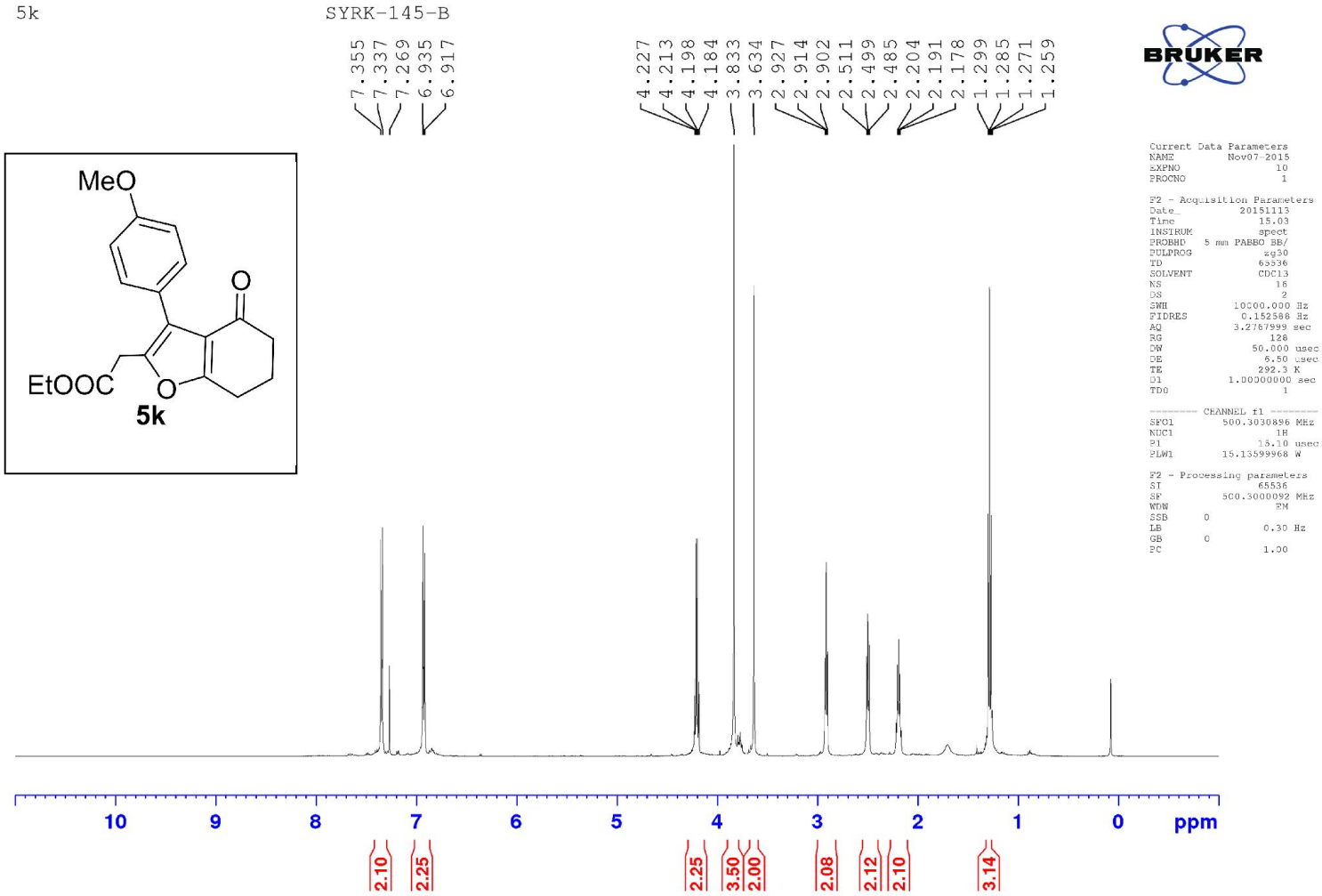

5i
SYRK81A
193.96
169.42
166.75
144.67
130.88
129.71
128.03
127.64
122.31
119.76
61.47
38.61
32.43
23.74
22.39
14.17
BRUKER
O
O
COOEt
(5i)
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

5j SYRK144A


7.325
7.313
7.309
7.225
7.209
4.244
4.229
4.215
4.201
3.658
2.951
2.938
2.925
2.533
2.521
2.507
2.239
2.226
2.213
2.200
2.188
1.316
1.301
1.287
BRUKER
O
O
COOEt
(5j)
Current Data Parameters
NAME Nov07-2015
EXPNO 92
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151108
Time 16.01
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 144
DW 50.000 usec
DE 6.50 usec
TE 292.1 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
SFO1 500.3030896 MHz
NUC1 1H
P1 15.10 usec
PLW1 15.13599968 W
F2 - Processing parameters
SI 65536
SF 500.3000000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
0
ppm
1.99
2.09
2.05
2.00
1.93
2.06
3.31
2.17
3.53

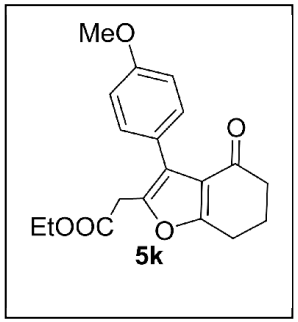
SYRK144A


193.95
169.48
166.67
144.44
137.34
129.56
128.79
127.85
122.24
119.81
61.44
38.63
32.43
23.76
22.40
21.32
14.18
BRUKER
O
O
COOEt
(5j)
Current Data Parameters
NAME Nov07-2015
EXPNO 93
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151108
Time 16.10
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 141
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 293.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
SFO1 125.8131145 MHz
NUC1 13C
P1 9.70 usec
PLW1 73.00000000 W
CHANNEL f2
SFO2 500.3020012 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 15.13599968 W
PLW12 0.53924000 W
PLW13 0.34512001 W
F2 - Processing parameters
SI 32768
SF 125.8005351 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

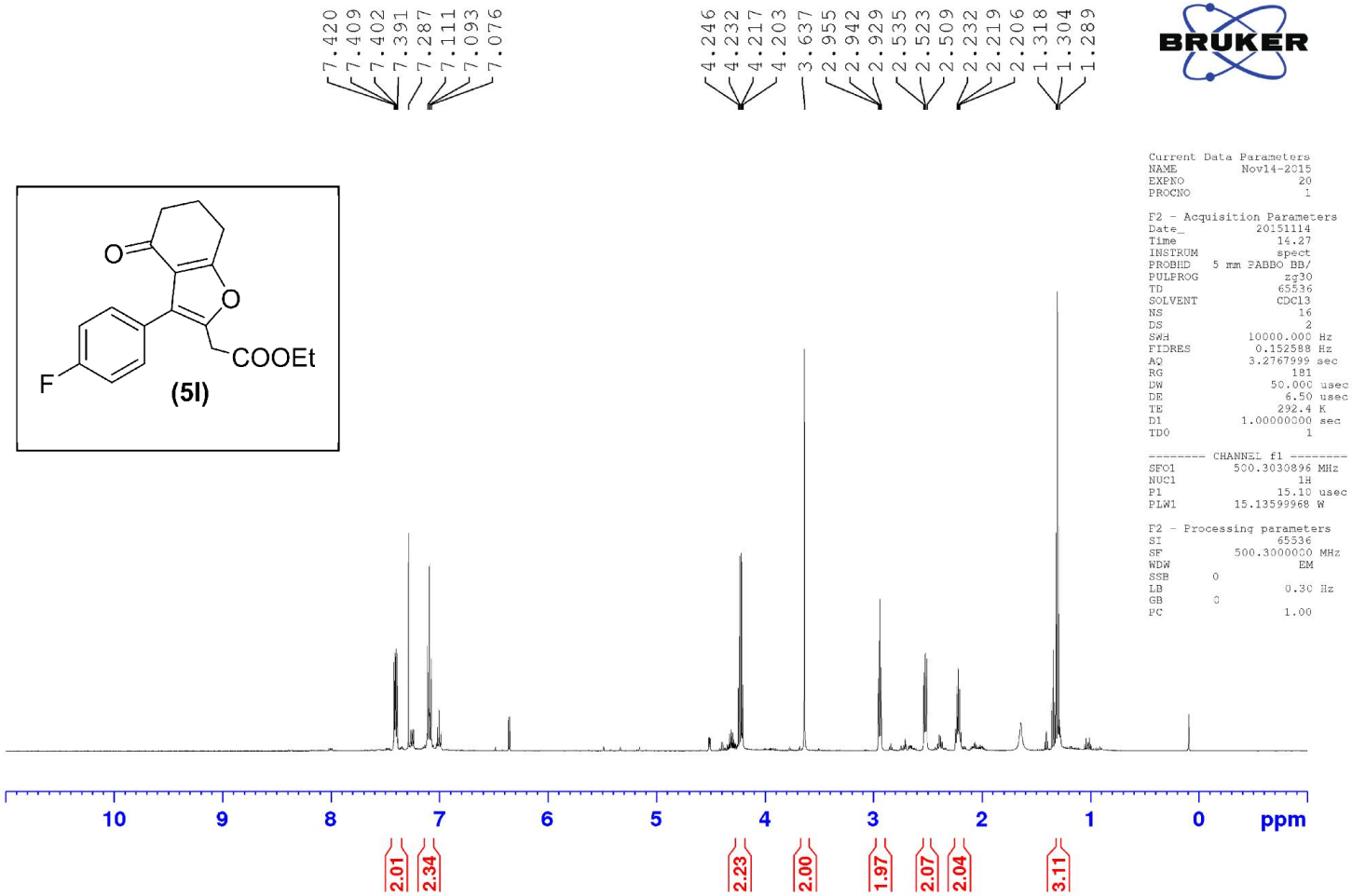

5k
SYRK-145-B
7.355
7.337
7.269
6.935
6.917
4.227
4.213
4.198
4.184
3.833
3.634
2.927
2.914
2.902
2.511
2.499
2.485
2.204
2.191
2.178
1.299
1.285
1.271
1.259
MeO
O
EtOOC
O
5k
BRUKER
Current Data Parameters
NAME Nov07-2015
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151113
Time 15.03
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 128
DW 50.000 usec
DE 6.50 usec
TE 292.3 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
SFO1 500.3030886 MHz
NUC1 1H
P1 13.10 usec
PLW1 15.13599968 W
F2 - Processing parameters
SI 65536
SF 500.3000092 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
0
ppm
2.10
2.25
2.25
3.50
2.00
2.08
2.12
2.10
3.14

SYRK-145B

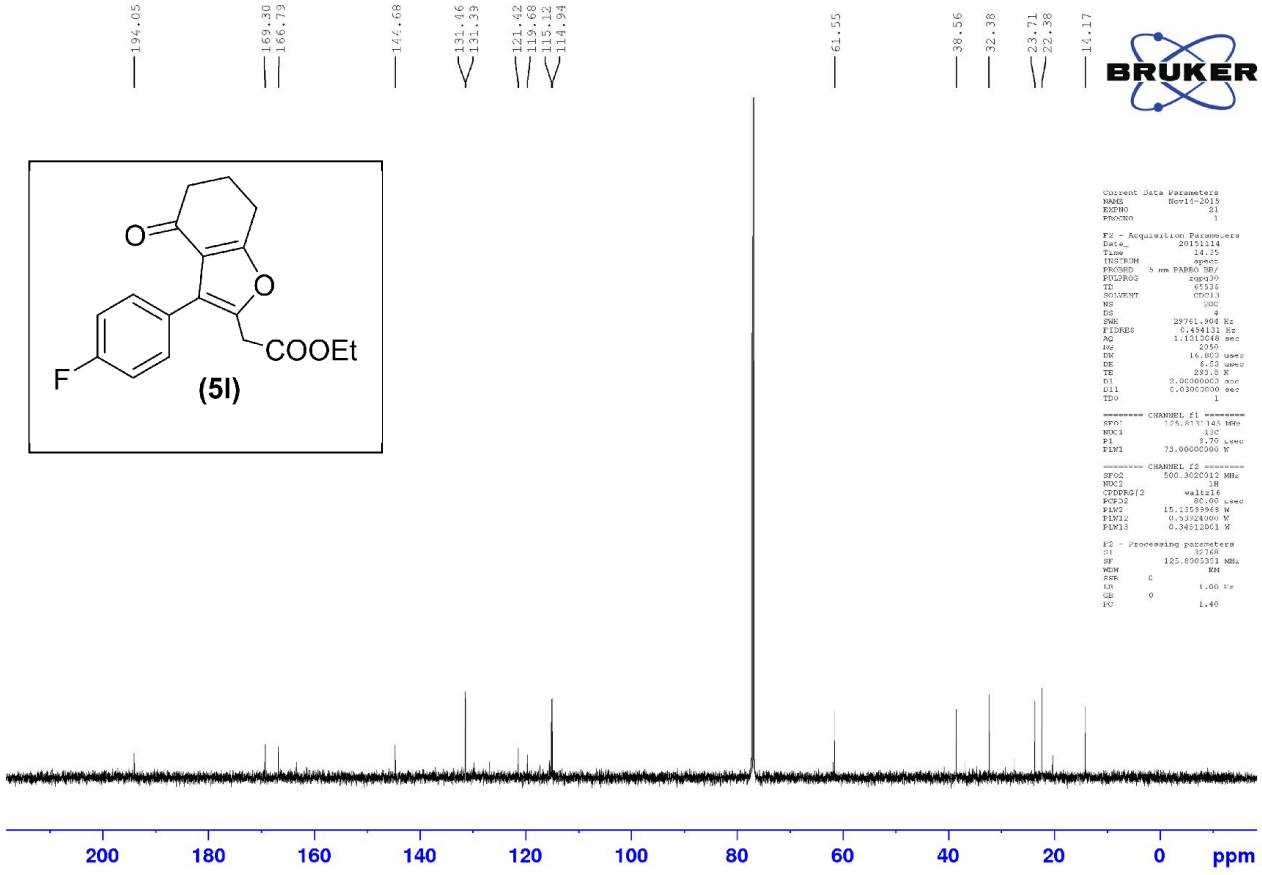
194.08
169.52
166.67
159.09
144.27
130.89
123.10
121.94
119.79
113.53
61.45
55.26
38.63
32.44
23.76
22.40
14.19



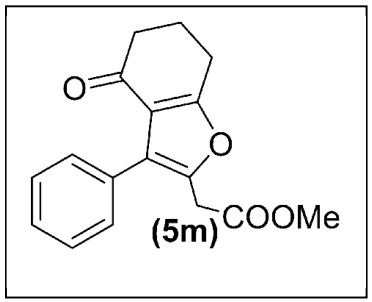

```
Current Data Parameters
NAME          Nov14 2015
EXPNO                 26
PROCNO                 1

F2 - Acquisition Parameters
Date_          20151114
Time              15.16
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   86
DS                    4
SWH           29761.904 Hz
FIDRES        0.454131 Hz
AQ            1.1010048 sec
RG                 2050
DW               16.800 usec
DE                6.50 usec
TE               293.5 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         125.8131145 MHz
NUC1               13c
P1                9.70 usec
PLW1         73.00000000 W

======== CHANNEL f2 ========
SFO2         500.3020012 MHz
NUC2               1H
CPDPRG[2         waltz16
PCPD2            80.00 usec
PLW2         15.13599968 W
PLW12         0.53924000 W
PLW13         0.34512001 W

F2 - Processing parameters
SI                32768
SF           125.8065351 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```


5l
SYRK-146B
7.420
7.409
7.402
7.391
7.287
7.111
7.093
7.076
4.246
4.232
4.217
4.203
3.637
2.955
2.942
2.929
2.535
2.523
2.509
2.232
2.219
2.206
1.318
1.304
1.289
BRUKER
O
O
COOEt
F
(5l)
Current Data Parameters
NAME Nov14-2015
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151114
Time 14.27
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 181
DW 50.000 usec
DE 6.50 usec
TE 292.4 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
SFO1 500.3030896 MHz
NUC1 1H
P1 15.10 usec
PLW1 15.13599968 W
F2 - Processing parameters
SI 65536
SF 500.3000000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
0
ppm
2.01
2.34
2.23
2.00
1.97
2.07
2.04
3.11

SYRK-146B

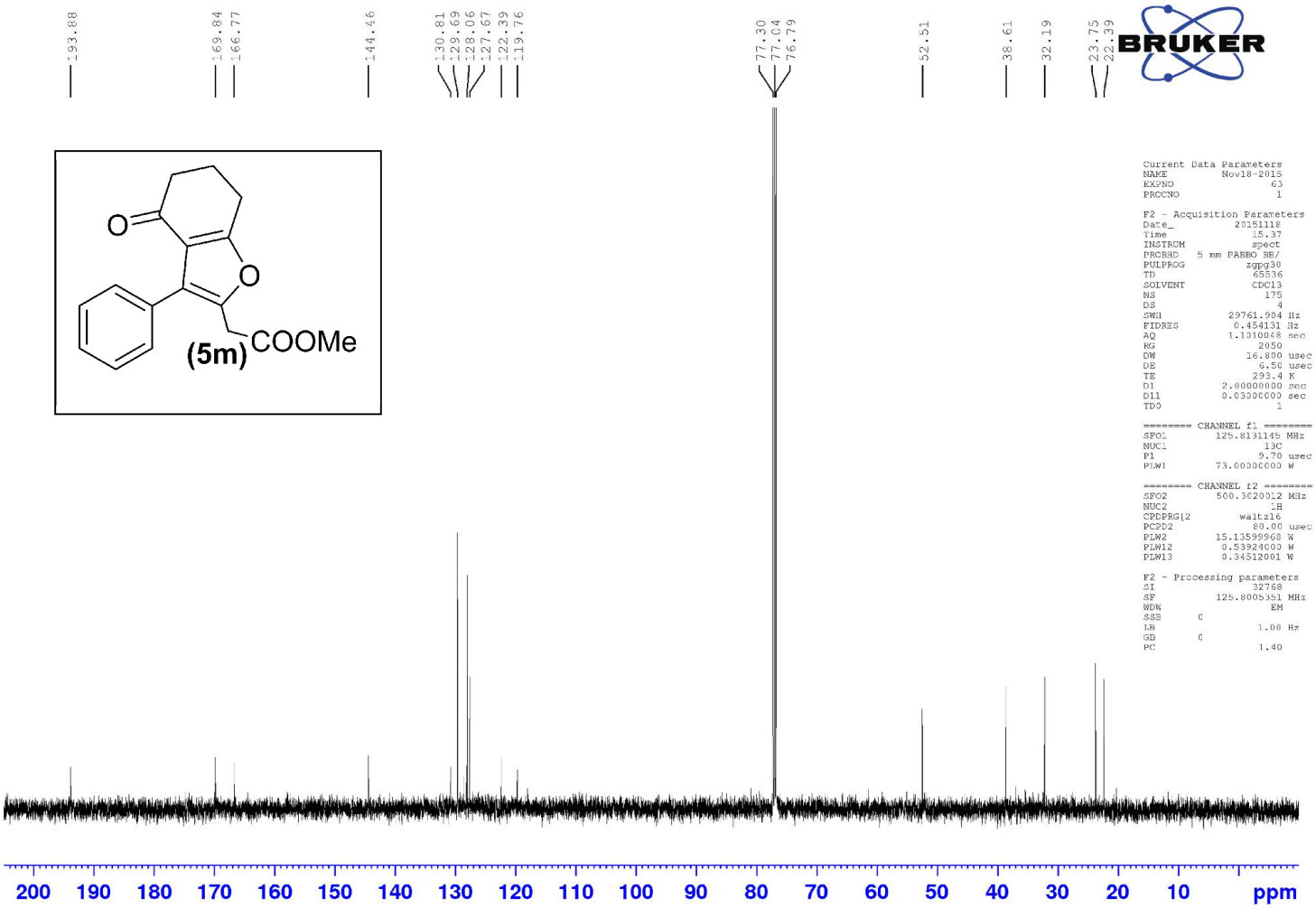

194.05
169.30
166.73
144.68
131.46
131.39
121.42
119.68
115.12
114.94
61.55
38.56
32.38
23.71
22.38
14.17
BRUKER
O
O
COOEt
F
(5l)
200
180
160
140
120
100
80
60
40
20
0
ppm

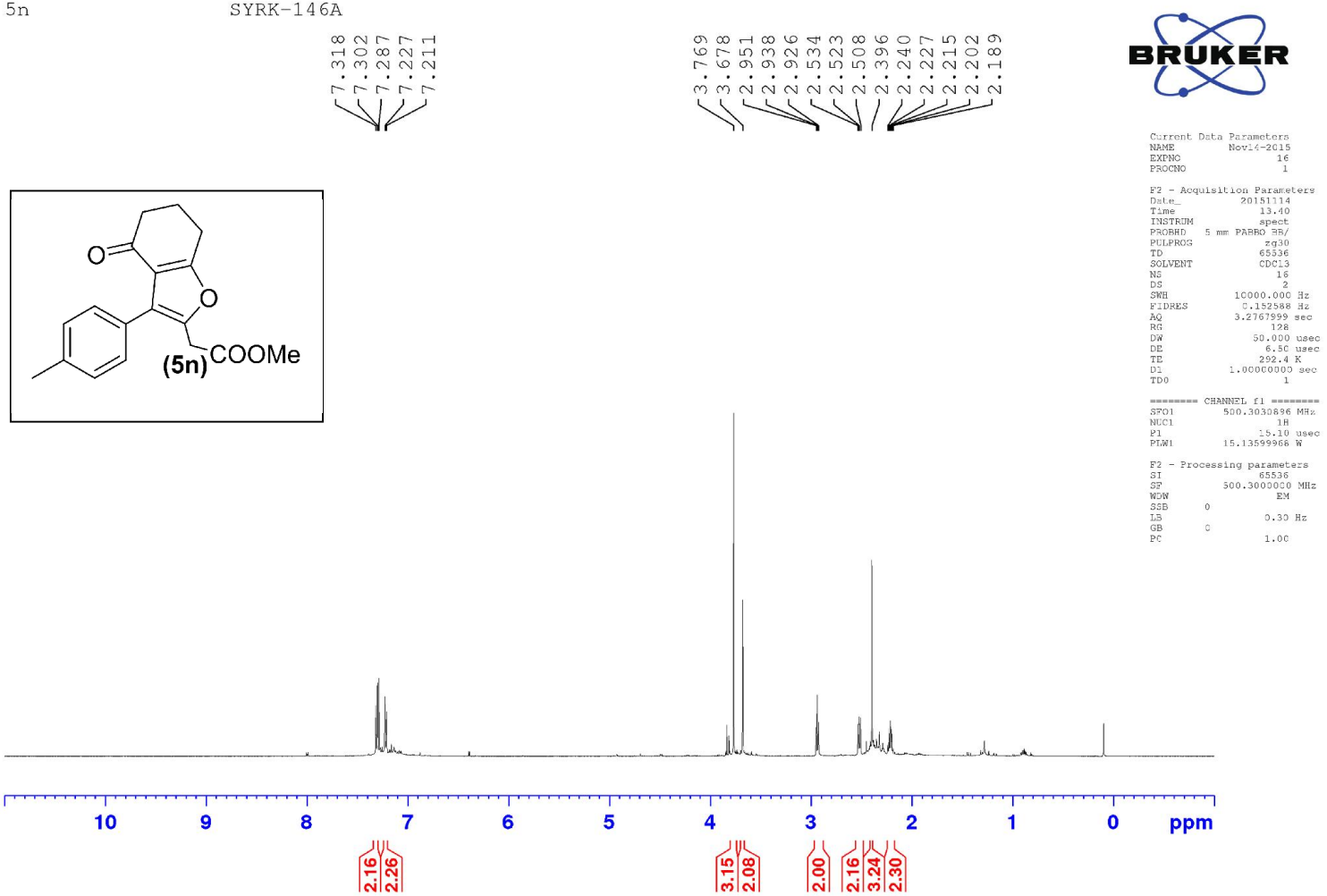
5m SYRK-153A

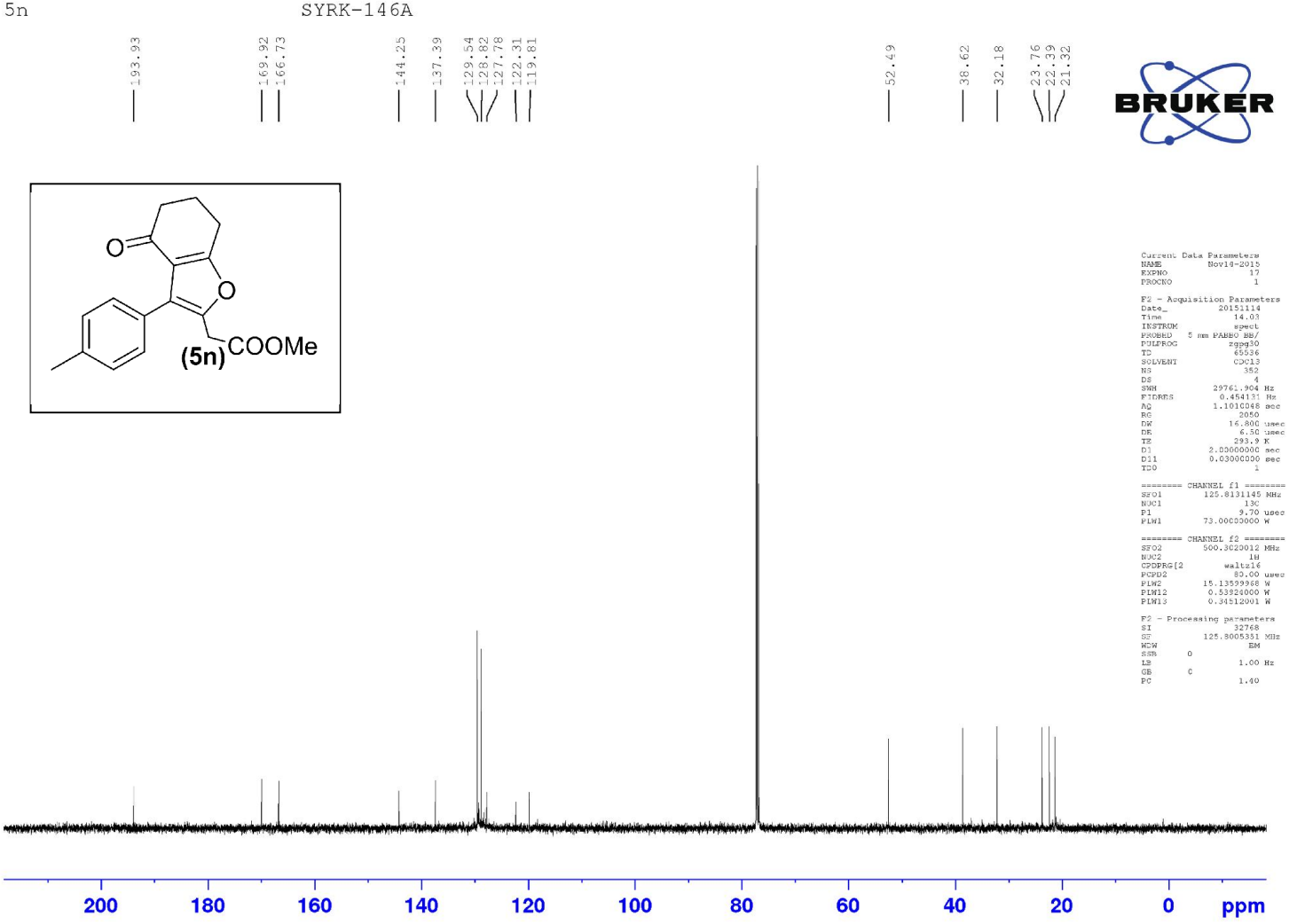



```
Current Data Parameters
NAME                  Nov18-2015
EXPNO                         62
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20151118
Time                       15.33
INSTRUM                    spect
PROBHD    5 mm PABBO BB/
PULPROG                    zg30
TD                        65536
SOLVENT                   CDCl3
NS                           16
DS                            2
SWH                   10000.000 Hz
FIDRES                0.152588 Hz
AQ                    3.2767999 sec
RG                          181
DW                       50.000 usec
DE                        6.50 usec
TE                       292.3 K
D1                    1.00000000 sec
TD0                            1

======== CHANNEL f1 ========
SFO1                500.3030896 MHz
NUC1                        1H
P1                       15.10 usec
PLW1                15.13599968 W

F2 - Processing parameters
SI                        65536
SF                  500.3000000 MHz
WDW                          EM
SSB                 0
LB                        0.30 Hz
GB                  0
PC                        1.00
```

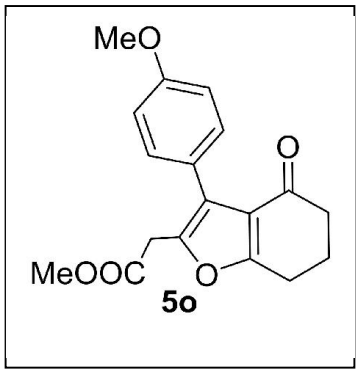
SYRK-153A


193.88
169.84
166.77
144.46
130.81
129.69
128.06
127.67
122.39
119.76
77.30
77.04
76.79
52.51
38.61
32.19
23.75
22.39
BRUKER
O
O
(5m)COOMe
Current Data Parameters
NAME Nov18-2015
EXPNO 63
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151118
Time 15.37
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 176
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 293.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.8131145 MHz
NUC1 13C
P1 9.70 usec
PLW1 73.00000000 W
======== CHANNEL f2 ========
SFO2 500.3620012 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 15.13599968 W
PLW12 0.53924000 W
PLW13 0.34512001 W
F2 - Processing parameters
SI 32768
SF 125.8005351 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


5n
SYRK-146A
7.318
7.302
7.287
7.227
7.211
3.769
3.678
2.951
2.938
2.926
2.534
2.523
2.508
2.396
2.240
2.227
2.215
2.202
2.189
BRUKER
Current Data Parameters
NAME Nov14-2015
EXPNO 16
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151114
Time 13.40
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 128
DW 50.000 usec
DE 6.50 usec
TE 292.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.3030896 MHz
NUC1 1H
P1 15.10 usec
PLW1 15.13599968 W
F2 - Processing parameters
SI 65536
SF 500.3000000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
O
(5n)COOMe
10
9
8
7
6
5
4
3
2
1
0
ppm
2.16
2.26
3.15
2.08
2.00
2.16
3.24
2.30

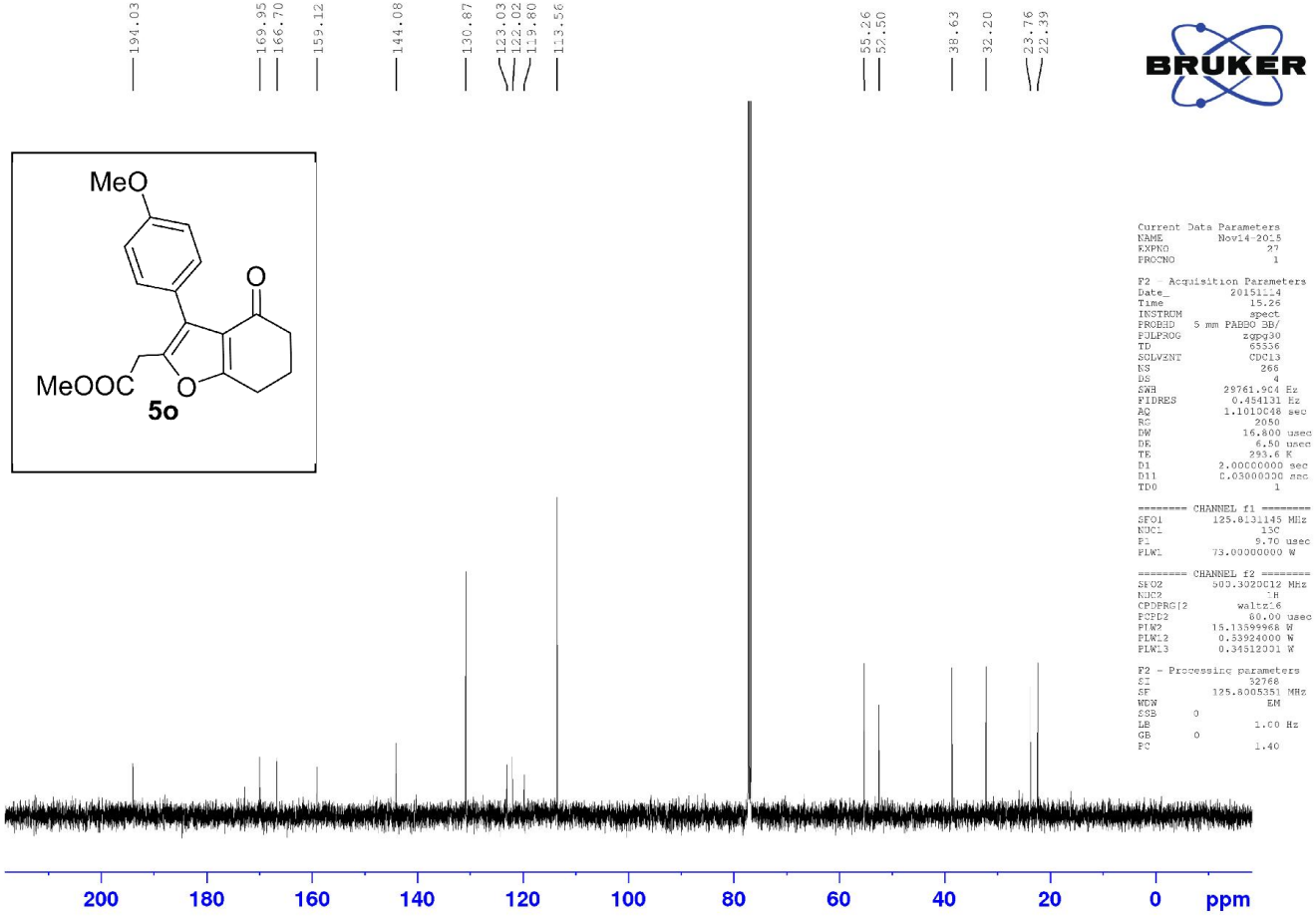

5n
SYRK-146A
193.93
169.92
166.73
144.25
137.39
129.54
128.82
127.78
122.31
119.81
52.49
38.62
32.18
23.76
22.39
21.32
BRUKER
O
O
(5n)COOMe
Current Data Parameters
NAME Nov14-2015
EXPNO 17
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151114
Time 14.03
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 352
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 293.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.8131145 MHz
NUC1 13C
P1 9.70 usec
PLW1 73.00000000 W
======== CHANNEL f2 ========
SFO2 500.3020012 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 15.13599968 W
PLW12 0.53924000 W
PLW13 0.34512001 W
F2 - Processing parameters
SI 32768
SF 125.8005351 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

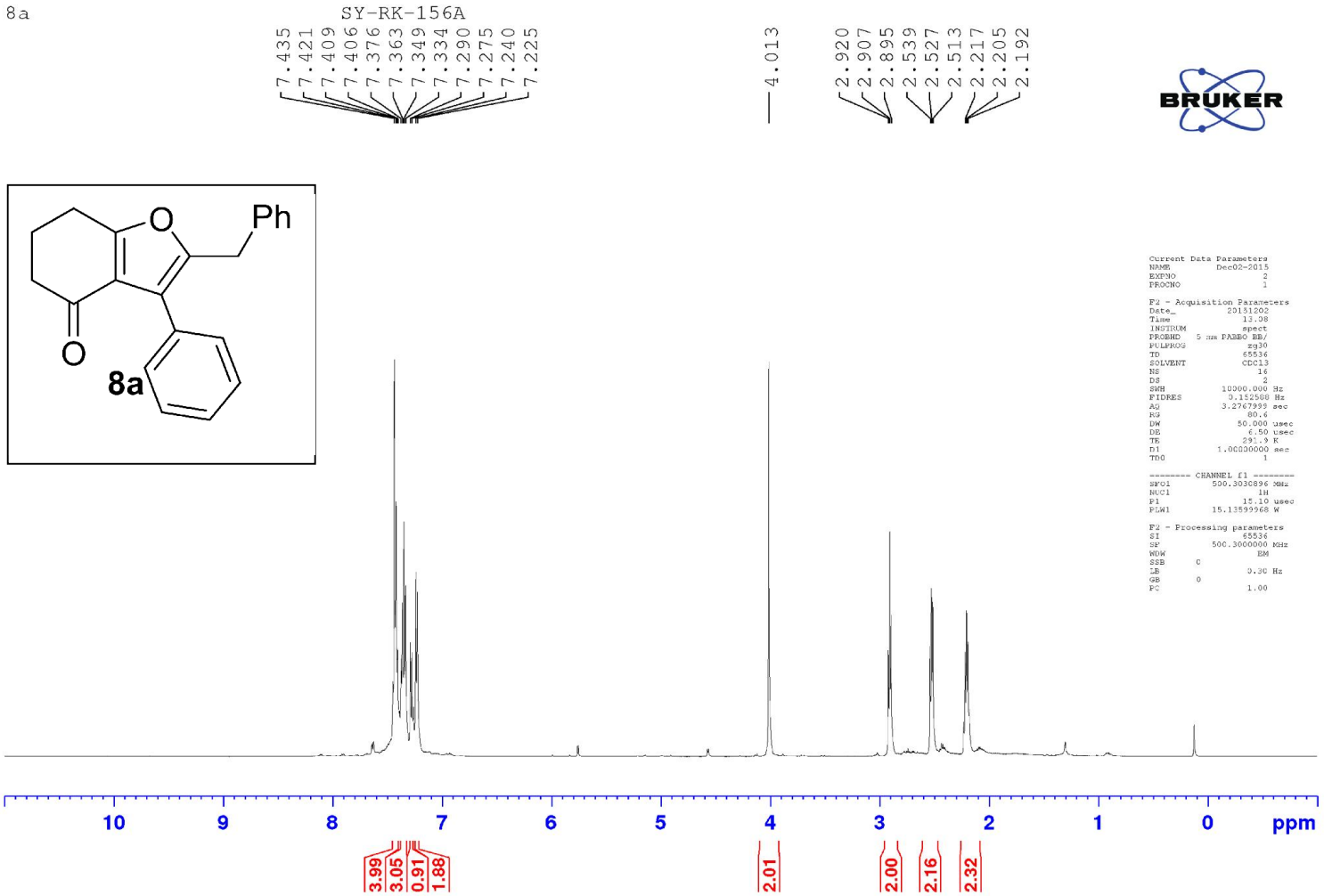
5o SYRK-147-A

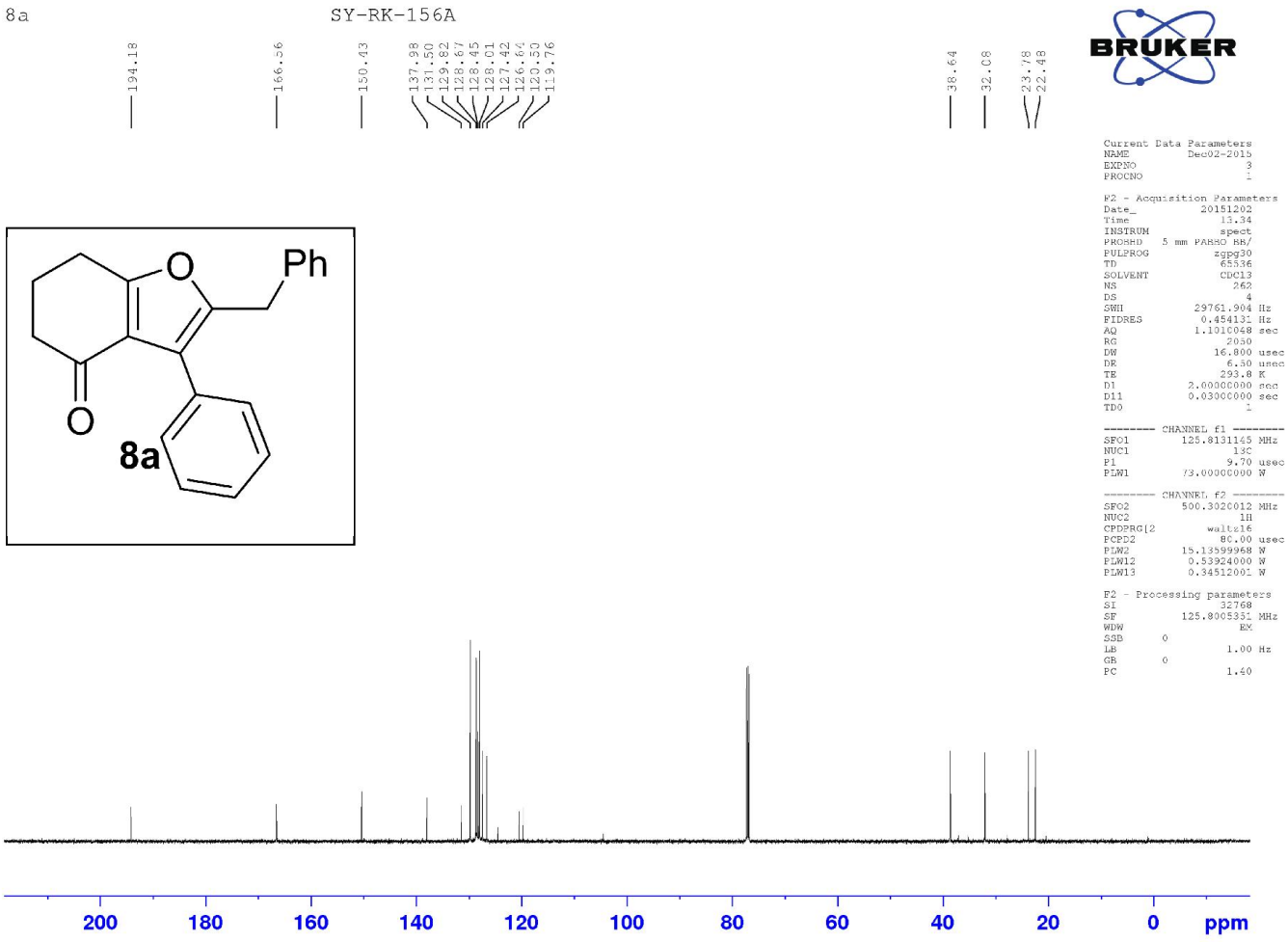

MeO
O
MeOOC
O
5o

7.347
7.330
7.269
6.939
6.921
3.836
3.755
3.654
2.930
2.917
2.905
2.515
2.503
2.489
2.208
2.195
2.183
0.076
BRUKER
Current Data Parameters
NAME Ncv07-2015
EXPNO 100
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151113
Time 15.08
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 203
DW 50.000 usec
DE 6.50 usec
TE 292.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.3030896 MHz
NUC1 1H
P1 15.10 usec
PLW1 15.13599968 W
F2 - Processing parameters
SI 65536
SF 500.3000092 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
0
ppm
1.94
2.12
3.16
3.34
2.00
2.00
2.06
2.03

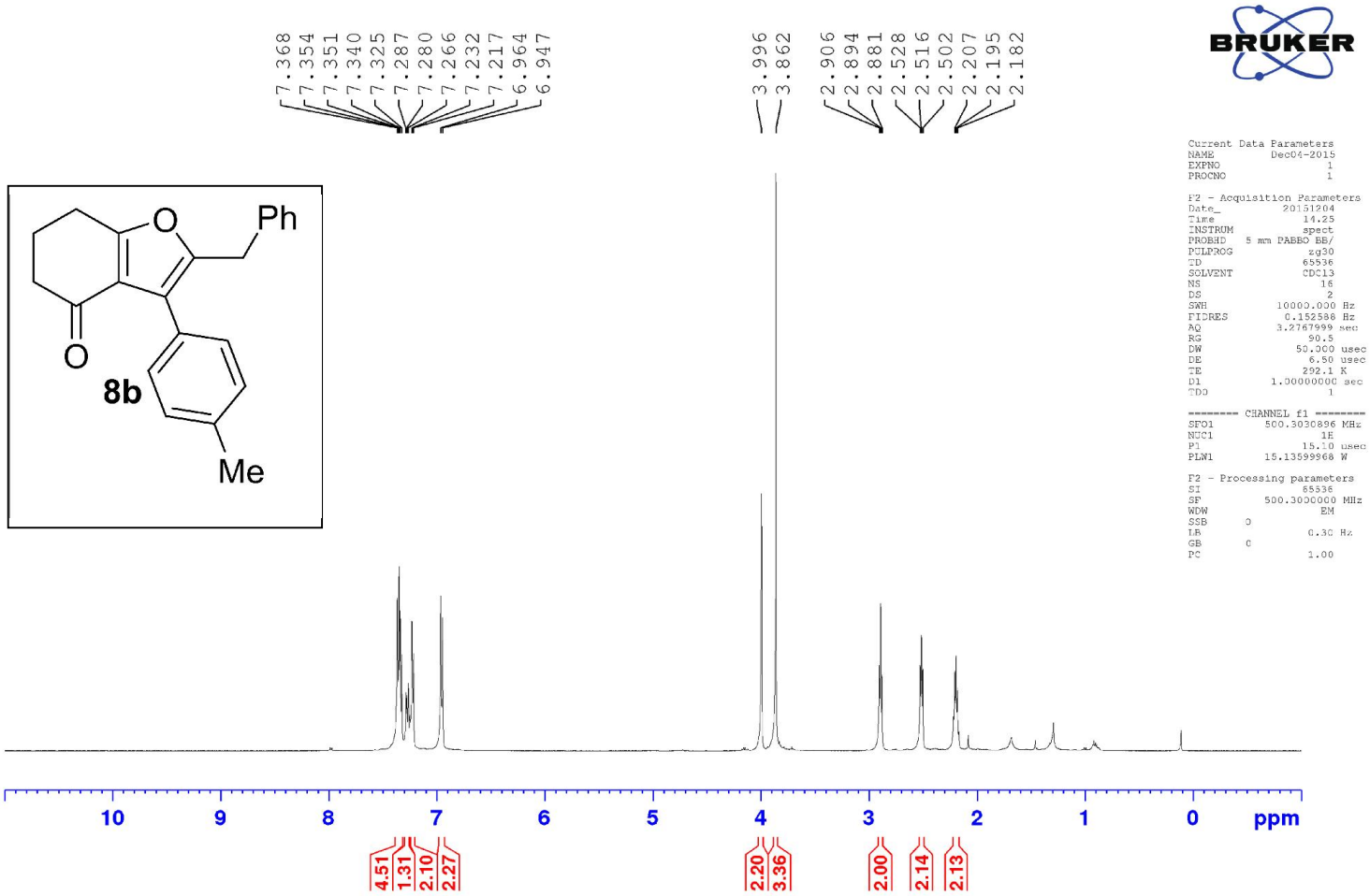
SYRK-147A


194.03
169.95
166.70
159.12
144.08
130.87
123.03
122.02
119.80
113.56
55.26
52.50
38.63
32.20
23.76
22.39
BRUKER
MeO
O
MeOOC
O
5o
Current Data Parameters
NAME Nov14-2015
EXPNO 27
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151124
Time 15.26
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 293.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.8131145 MHz
NUC1 13C
P1 9.70 usec
PLW1 73.00000000 W
======== CHANNEL f2 ========
SFO2 500.3020012 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 60.00 usec
PLW2 15.13599968 W
PLW12 0.53924000 W
PLW13 0.34812001 W
F2 - Processing parameters
SI 32768
SF 125.8005351 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm


8a
SY-RK-156A
7.435
7.421
7.409
7.406
7.376
7.363
7.349
7.334
7.290
7.275
7.240
7.225
4.013
2.920
2.907
2.895
2.539
2.527
2.513
2.217
2.205
2.192
BRUKER
O
Ph
O
8a
Current Data Parameters
NAME Dec02-2015
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151202
Time 13.08
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767993 sec
RG 80.6
DW 50.000 usec
DE 6.50 usec
TE 291.9 K
D1 1.00000000 sec
TD0 1
-------- CHANNEL f1 --------
SFO1 500.3030896 MHz
NUC1 1H
P1 15.10 usec
PLW1 15.13599968 W
F2 - Processing parameters
SI 65536
SF 500.3000000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
0
ppm
3.99
3.05
0.91
1.88
2.01
2.00
2.16
2.32


8a
SY-RK-156A
194.18
166.56
150.43
137.98
131.50
129.82
128.67
128.45
128.01
127.42
126.64
120.50
119.76
38.64
32.08
23.78
22.48
BRUKER
O
Ph
O
8a
Current Data Parameters
NAME Dec02-2015
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151202
Time 13.34
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 262
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 293.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
SFO1 125.8131145 MHz
NUC1 13C
P1 9.70 usec
PLW1 73.00000000 W
CHANNEL f2
SFO2 500.3020012 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 15.13599968 W
PLW12 0.53924000 W
PLW13 0.34512001 W
F2 - Processing parameters
SI 32768
SF 125.8005351 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

8b SY-AP-155B

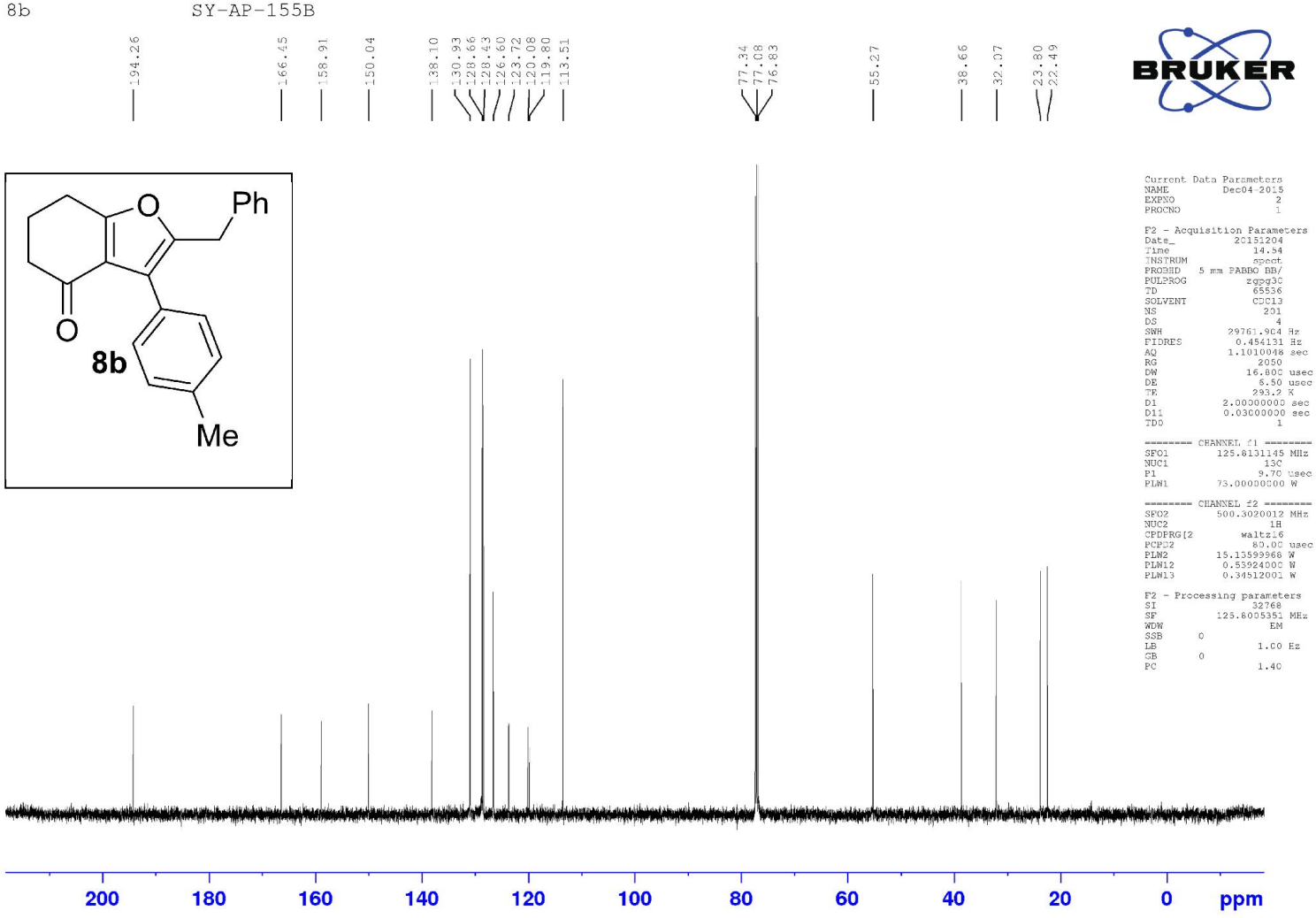


Current Data Parameters
NAME Dec04-2015
EXPNO 1
PROCNO 1

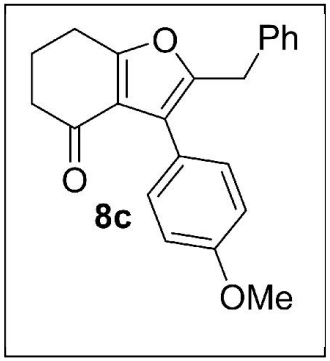
F2 - Acquisition Parameters
Date_ 20151204
Time 14.25
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 90.5
DW 50.000 usec
DE 6.50 usec
TE 292.1 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 500.3030896 MHz
NUC1 1H
P1 15.10 usec
PLW1 15.13599968 W

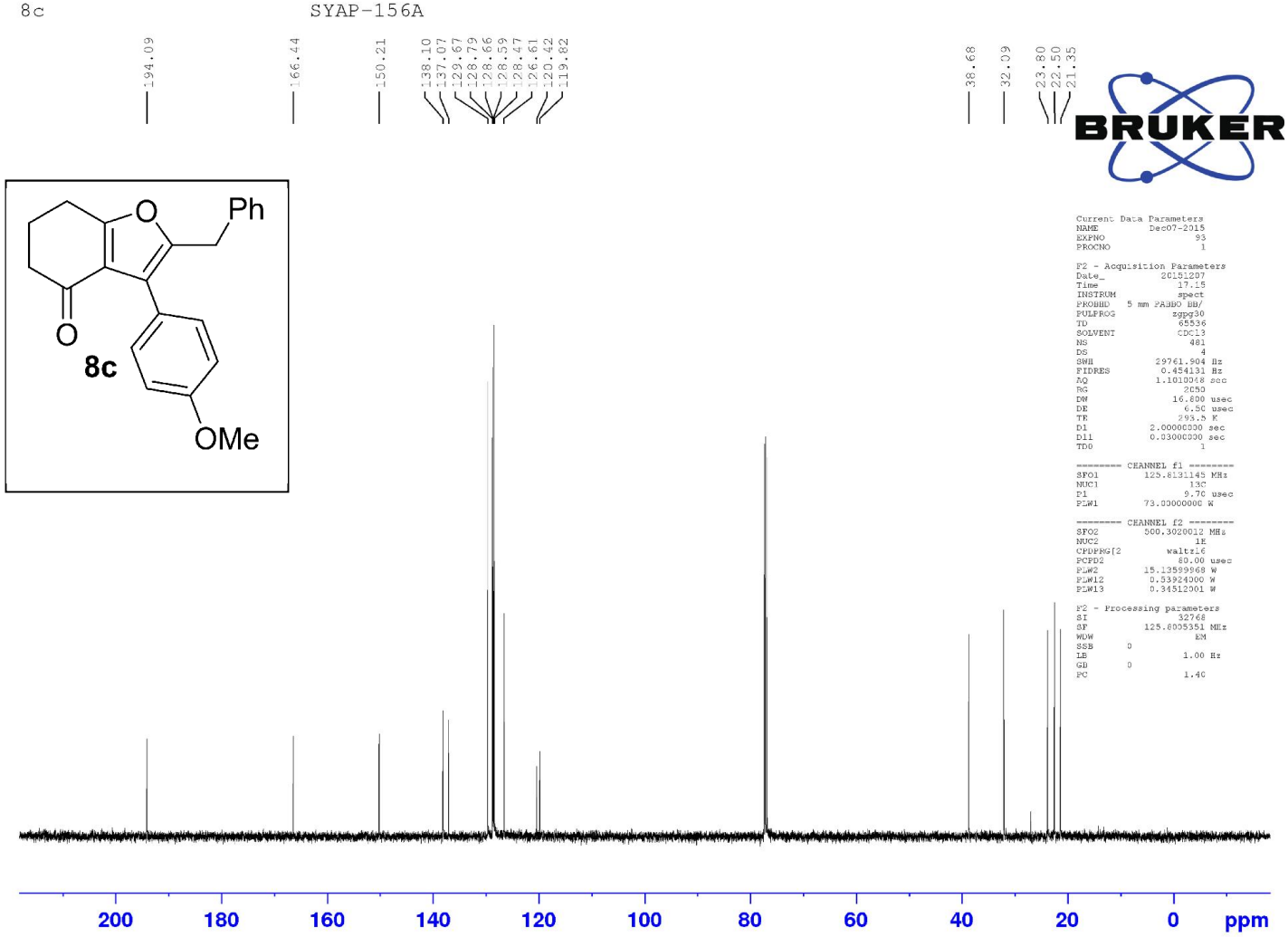
F2 - Processing parameters
SI 65536
SF 500.3000000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


8b
SY-AP-155B
194.26
166.45
158.91
150.04
138.10
130.93
128.66
128.43
126.60
123.72
120.08
119.80
113.51
77.34
77.08
76.83
55.27
38.66
32.07
23.80
22.49
BRUKER
O
Ph
O
8b
Me
Current Data Parameters
NAME Dec04-2015
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151204
Time 14.54
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 201
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 293.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.8131145 MHz
NUC1 13C
P1 9.70 usec
PLW1 73.00000000 W
======== CHANNEL f2 ========
SFO2 500.3020012 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 15.13599968 W
PLW12 0.55924000 W
PLW13 0.34512001 W
F2 - Processing parameters
SI 32768
SF 125.8005351 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

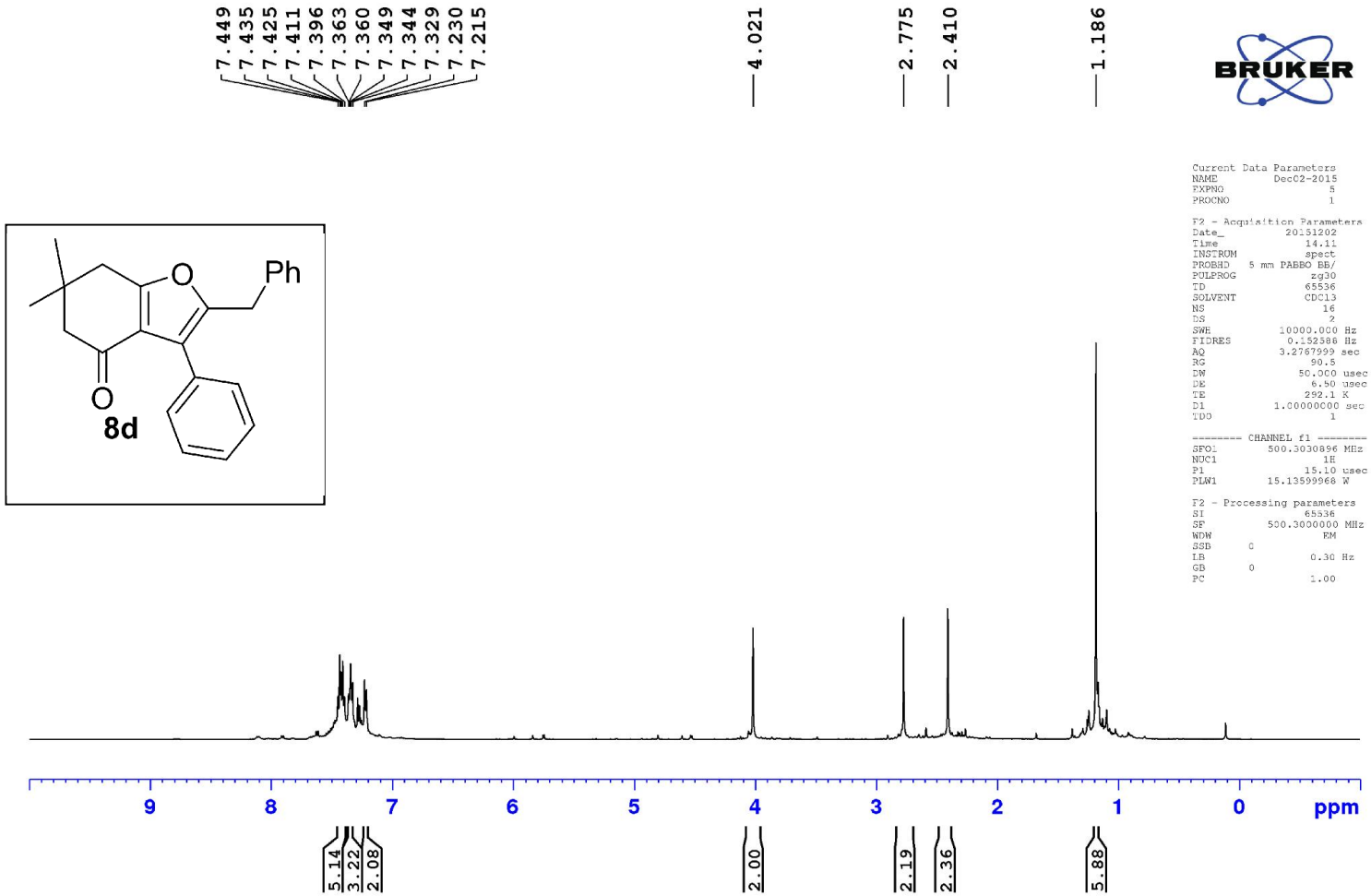
8c SYAP-156A

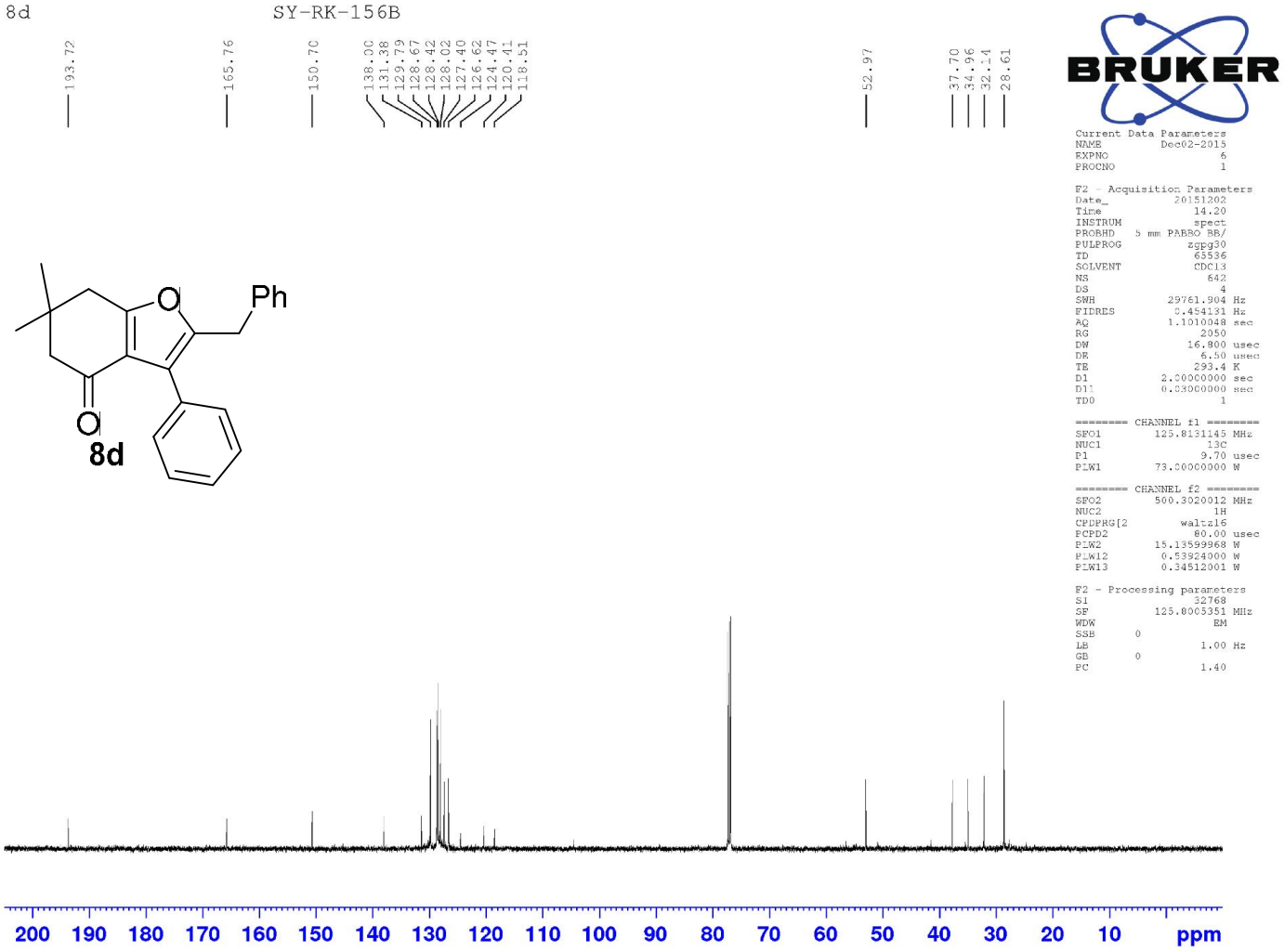

O
Ph
O
8c
OMe

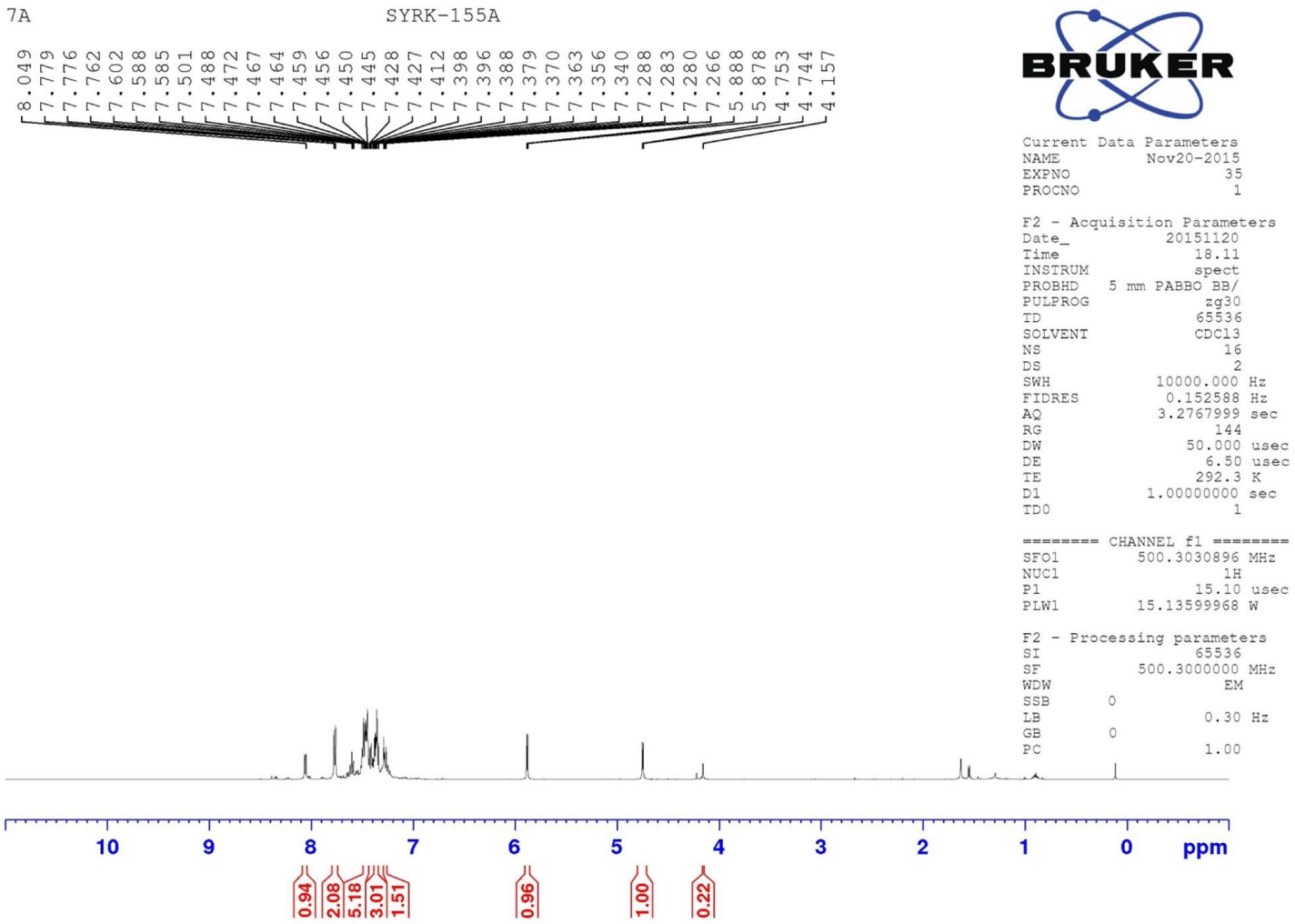
7.368
7.352
7.336
7.292
7.289
7.278
7.262
7.235
4.013
2.916
2.903
2.891
2.541
2.529
2.515
2.424
2.217
2.204
2.191
BRUKER
Current Data Parameters
NAME Dec07-2015
EXPNO 92
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151207
Time 17.38
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 64
DW 50.000 usec
DE 6.50 usec
TE 292.3 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
SFO1 500.3030896 MHz
NUC1 1H
P1 15.10 usec
PLW1 15.13599968 W
F2 - Processing parameters
SI 65536
SF 500.3000000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
0
ppm
4.09
1.05
3.97
2.00
1.95
2.00
2.95
2.11


8c
SYAP-156A
194.09
166.44
150.21
138.10
137.07
129.67
128.79
128.66
128.59
128.47
126.61
120.42
119.82
38.68
32.09
23.80
22.50
21.35
BRUKER
O
Ph
O
8c
OMe
Current Data Parameters
NAME Dec07-2015
EXPNO 93
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151207
Time 17.15
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 481
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 293.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.8133145 MHz
NUC1 13C
P1 9.70 usec
PLW1 73.00000000 W
======== CHANNEL f2 ========
SFO2 500.3020012 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 15.13599968 W
PLW12 0.53924000 W
PLW13 0.34512001 W
F2 - Processing parameters
SI 32768
SF 125.8005351 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

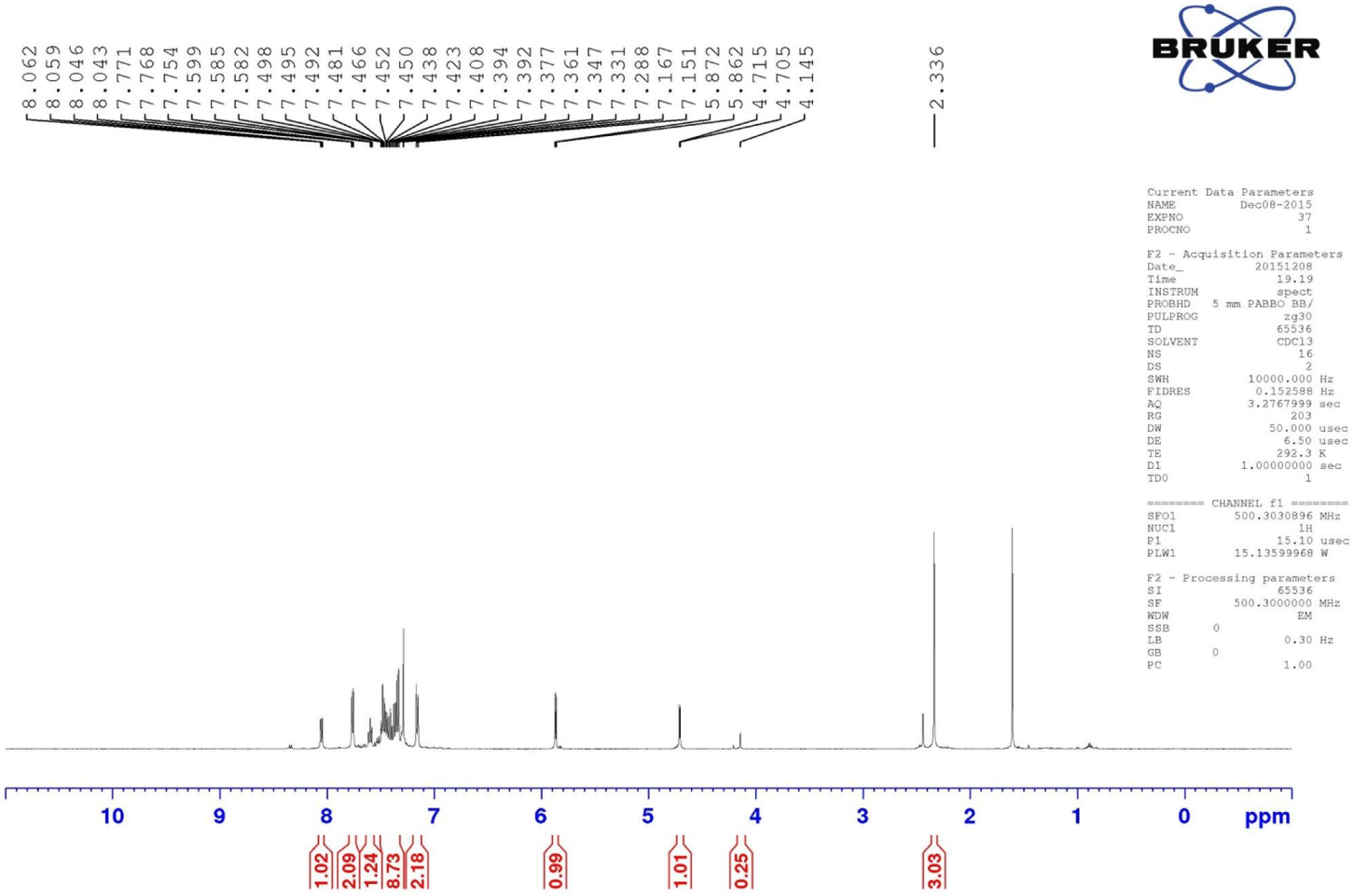
SY-RK-156B

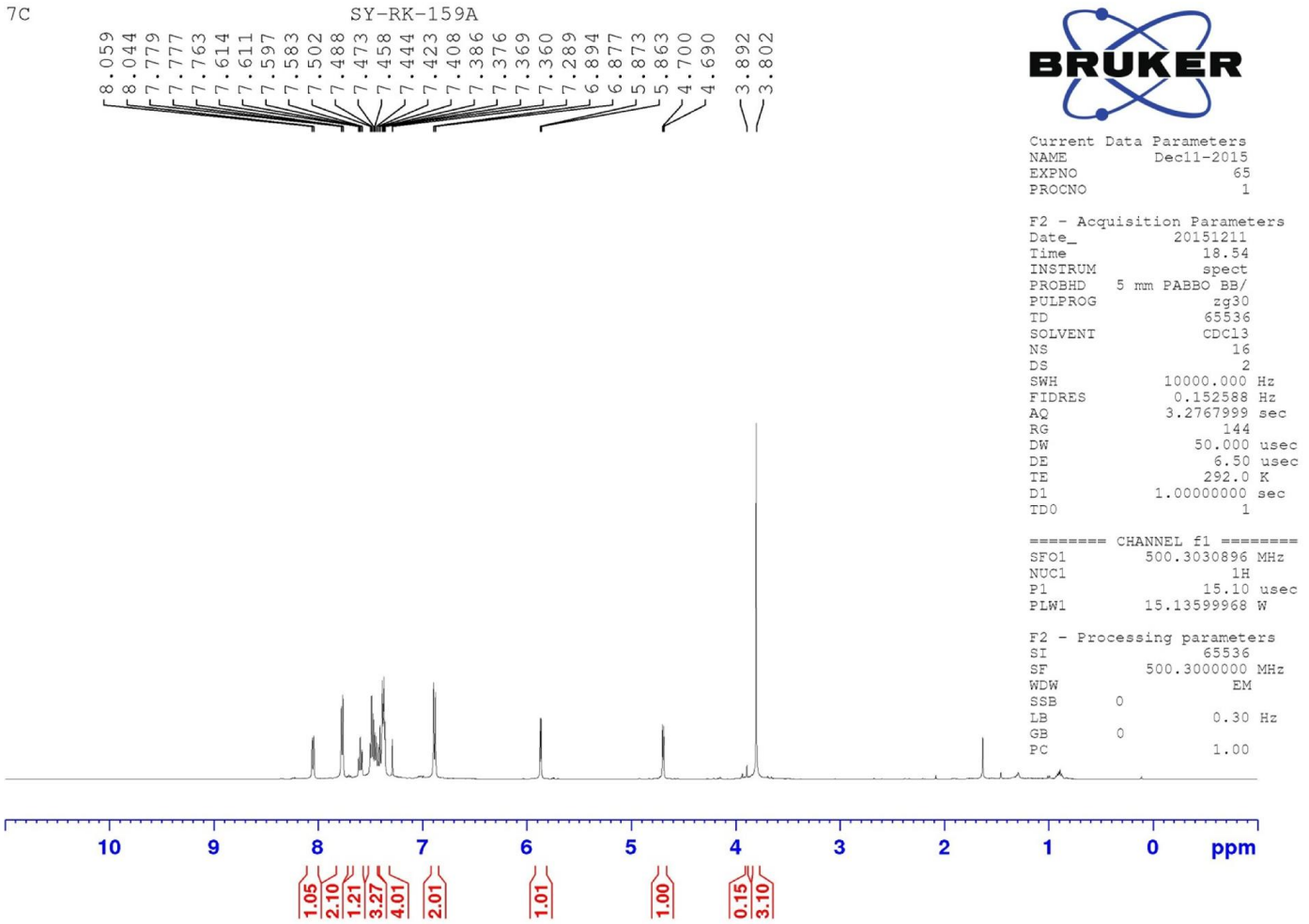

7.449
7.435
7.425
7.411
7.396
7.363
7.360
7.349
7.344
7.329
7.230
7.215
4.021
2.775
2.410
1.186
BRUKER
O
Ph
O
8d
Current Data Parameters
NAME DecC2-2015
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151202
Time 14.11
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152586 Hz
AQ 3.2767999 sec
RG 90.5
DW 50.000 usec
DE 6.50 usec
TE 292.1 K
D1 1.00000000 sec
TDO 1
======== CHANNEL f1 ========
SFO1 500.3030896 MHz
NUC1 1H
P1 15.10 usec
PLW1 15.13599968 W
F2 - Processing parameters
SI 65536
SF 500.3000000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0
ppm
5.14
3.22
2.08
2.00
2.19
2.36
5.88


8d
SY-RK-156B
193.72
165.76
150.70
138.00
131.38
129.79
128.67
128.42
128.02
127.40
126.62
124.47
120.41
118.51
52.97
37.70
34.96
32.14
28.61
BRUKER
Current Data Parameters
NAME Dec02-2015
EXPNO 6
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151202
Time 14.20
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 642
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 293.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.8131145 MHz
NUC1 13C
P1 9.70 usec
PLW1 73.00000000 W
======== CHANNEL f2 ========
SFO2 500.3020012 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 15.13599968 W
PLW12 0.53924000 W
PLW13 0.34512001 W
F2 - Processing parameters
SI 32768
SF 125.8005351 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Ph
O
O
8d
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

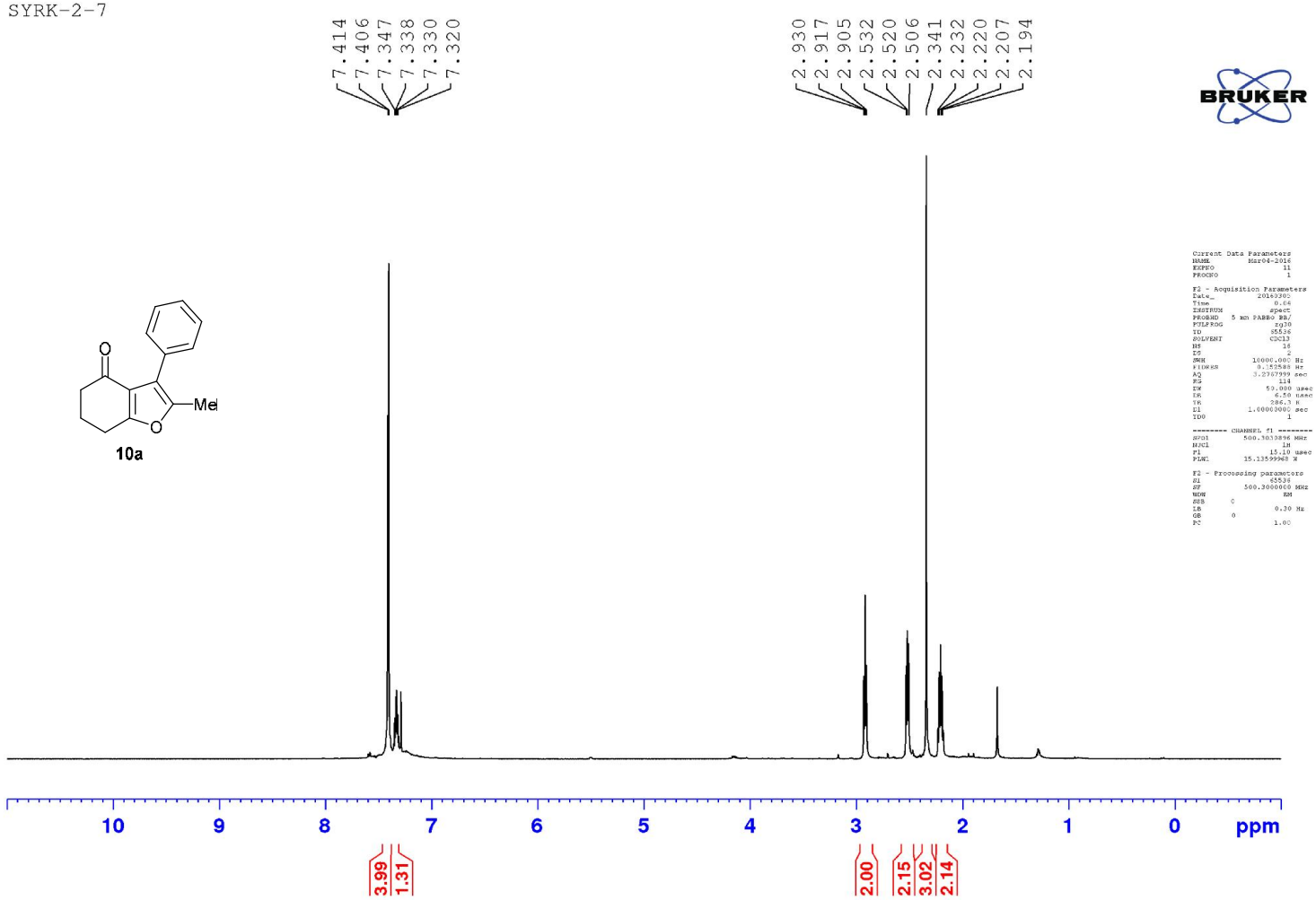

7A
SYRK-155A
8.049
7.779
7.776
7.762
7.602
7.588
7.585
7.501
7.488
7.472
7.467
7.464
7.459
7.456
7.450
7.445
7.428
7.427
7.412
7.398
7.396
7.388
7.379
7.370
7.363
7.356
7.340
7.288
7.283
7.280
7.266
5.888
5.878
4.753
4.744
4.157
BRUKER
Current Data Parameters
NAME Nov20-2015
EXPNO 35
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151120
Time 18.11
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 144
DW 50.000 usec
DE 6.50 usec
TE 292.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.3030896 MHz
NUC1 1H
P1 15.10 usec
PLW1 15.13599968 W
F2 - Processing parameters
SI 65536
SF 500.3000000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
0
ppm
0.94
2.08
5.18
3.01
1.51
0.96
1.00
0.22

7B SYRK-158B

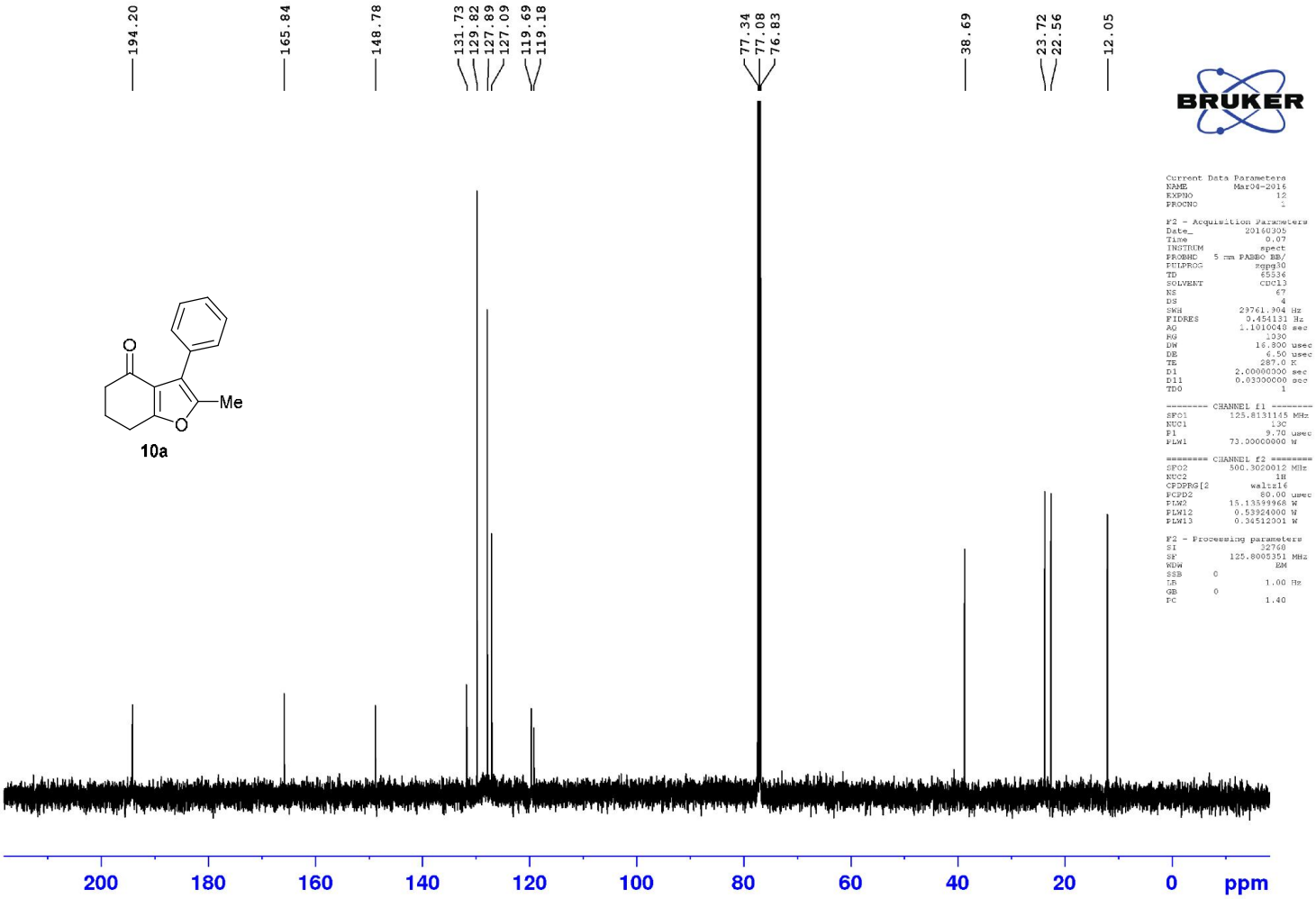

8.062
8.059
8.046
8.043
7.771
7.768
7.754
7.599
7.585
7.582
7.498
7.495
7.492
7.481
7.466
7.452
7.450
7.438
7.423
7.408
7.394
7.392
7.377
7.361
7.347
7.331
7.288
7.167
7.151
5.872
5.862
4.715
4.705
4.145
2.336
BRUKER
Current Data Parameters
NAME Dec08-2015
EXPNO 37
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151208
Time 19.19
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 203
DW 50.000 usec
DE 6.50 usec
TE 292.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.3030896 MHz
NUC1 1H
P1 15.10 usec
PLW1 15.13599968 W
F2 - Processing parameters
SI 65536
SF 500.3000000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
0
ppm
1.02
2.09
1.24
8.73
2.18
0.99
1.01
0.25
3.03


7C
SY-RK-159A
8.059
8.044
7.779
7.777
7.763
7.614
7.611
7.597
7.583
7.502
7.488
7.473
7.458
7.444
7.423
7.408
7.386
7.376
7.369
7.360
7.289
6.894
6.877
5.873
5.863
4.700
4.690
3.892
3.802
BRUKER
Current Data Parameters
NAME Dec11-2015
EXPNO 65
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151211
Time 18.54
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 144
DW 50.000 usec
DE 6.50 usec
TE 292.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.3030896 MHz
NUC1 1H
P1 15.10 usec
PLW1 15.13599968 W
F2 - Processing parameters
SI 65536
SF 500.3000000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
0
ppm
1.05
2.10
1.21
3.27
4.01
2.01
1.01
1.00
0.15
3.10

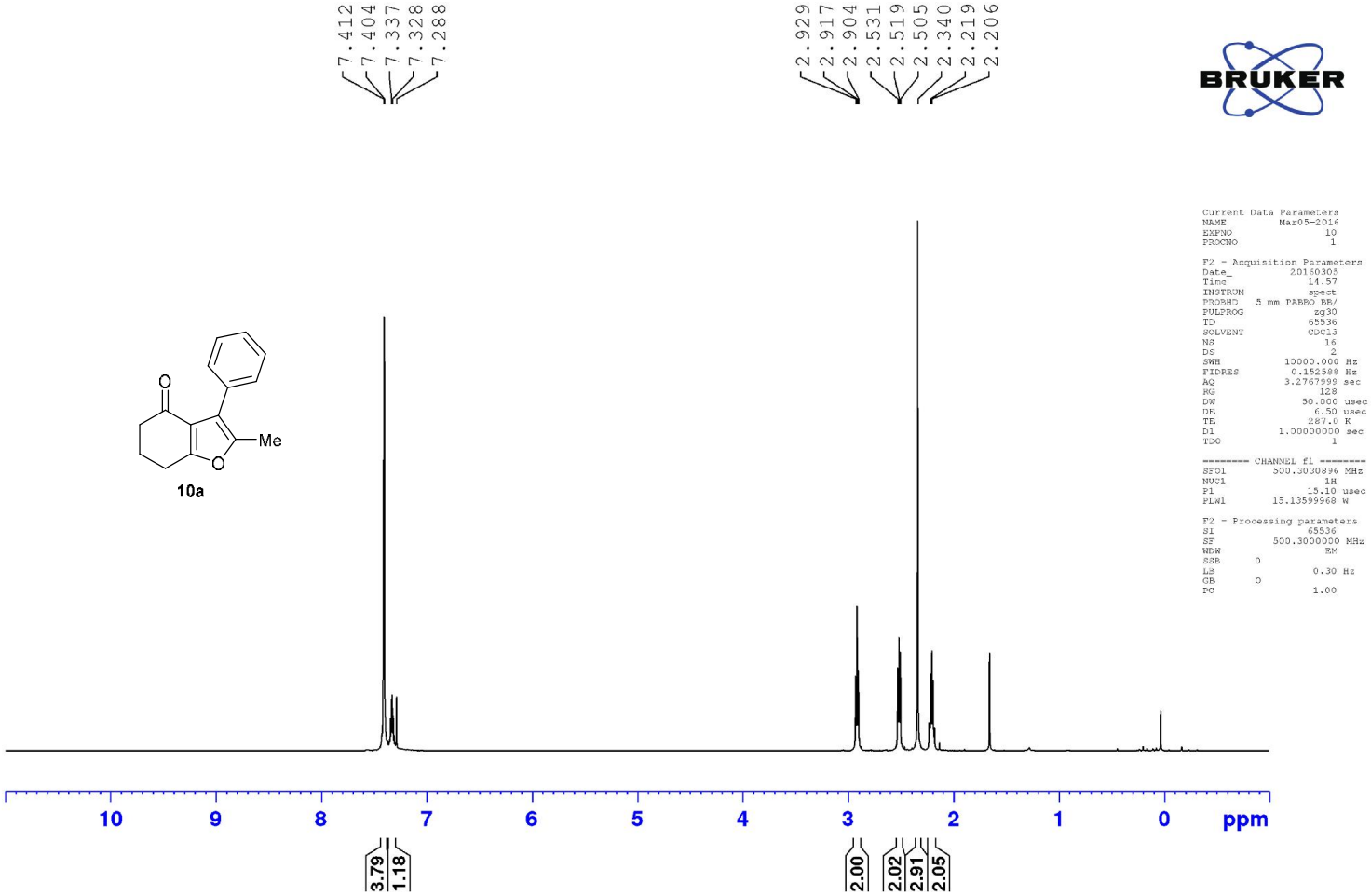
SYRK-2-7


7.414
7.406
7.347
7.338
7.330
7.320
2.930
2.917
2.905
2.532
2.520
2.506
2.341
2.232
2.220
2.207
2.194
BRUKER
O
Me
O
10a
10
9
8
7
6
5
4
3
2
1
0
ppm
3.99
1.31
2.00
2.15
3.02
2.14

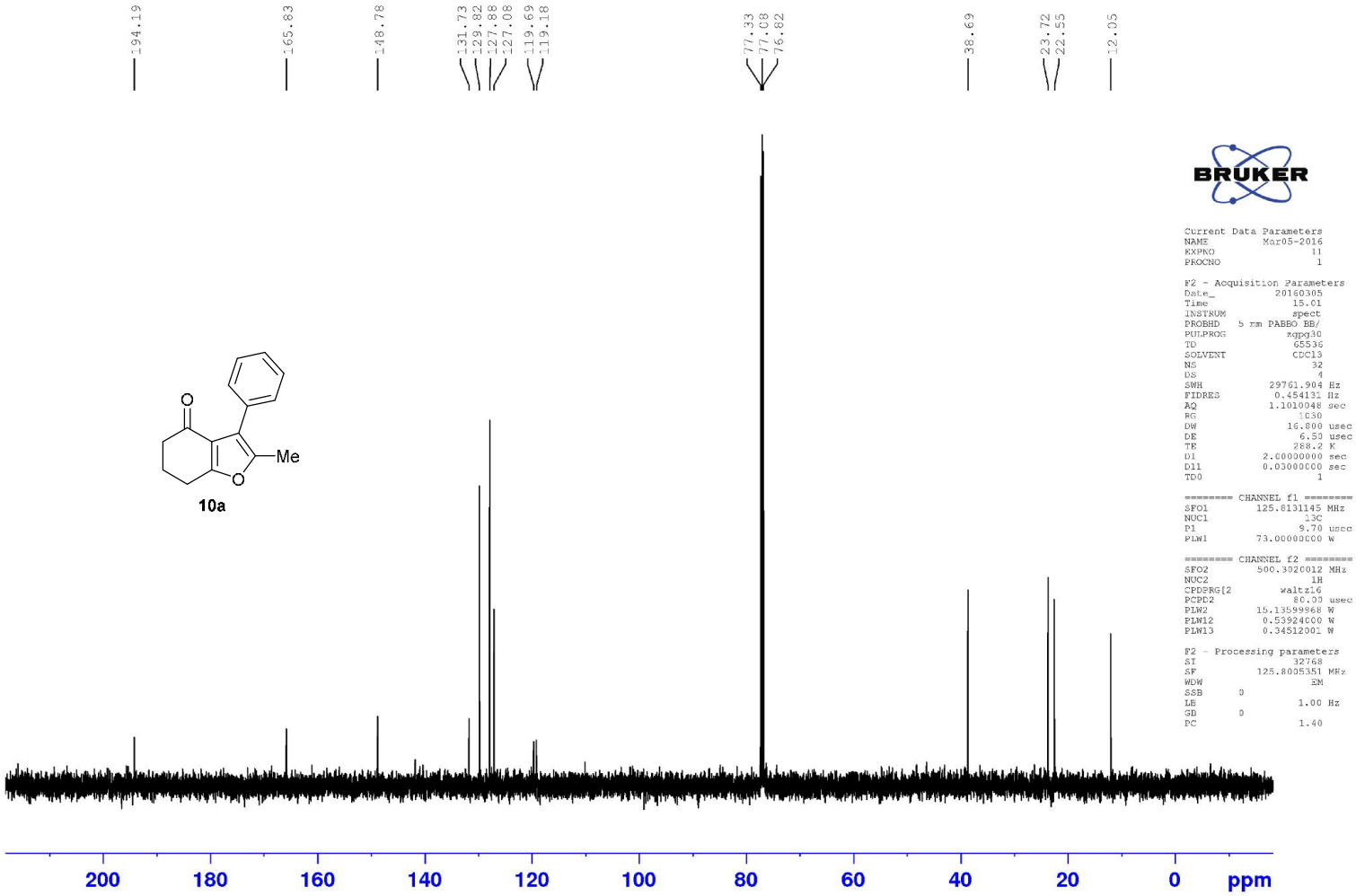
SYRK-2-7

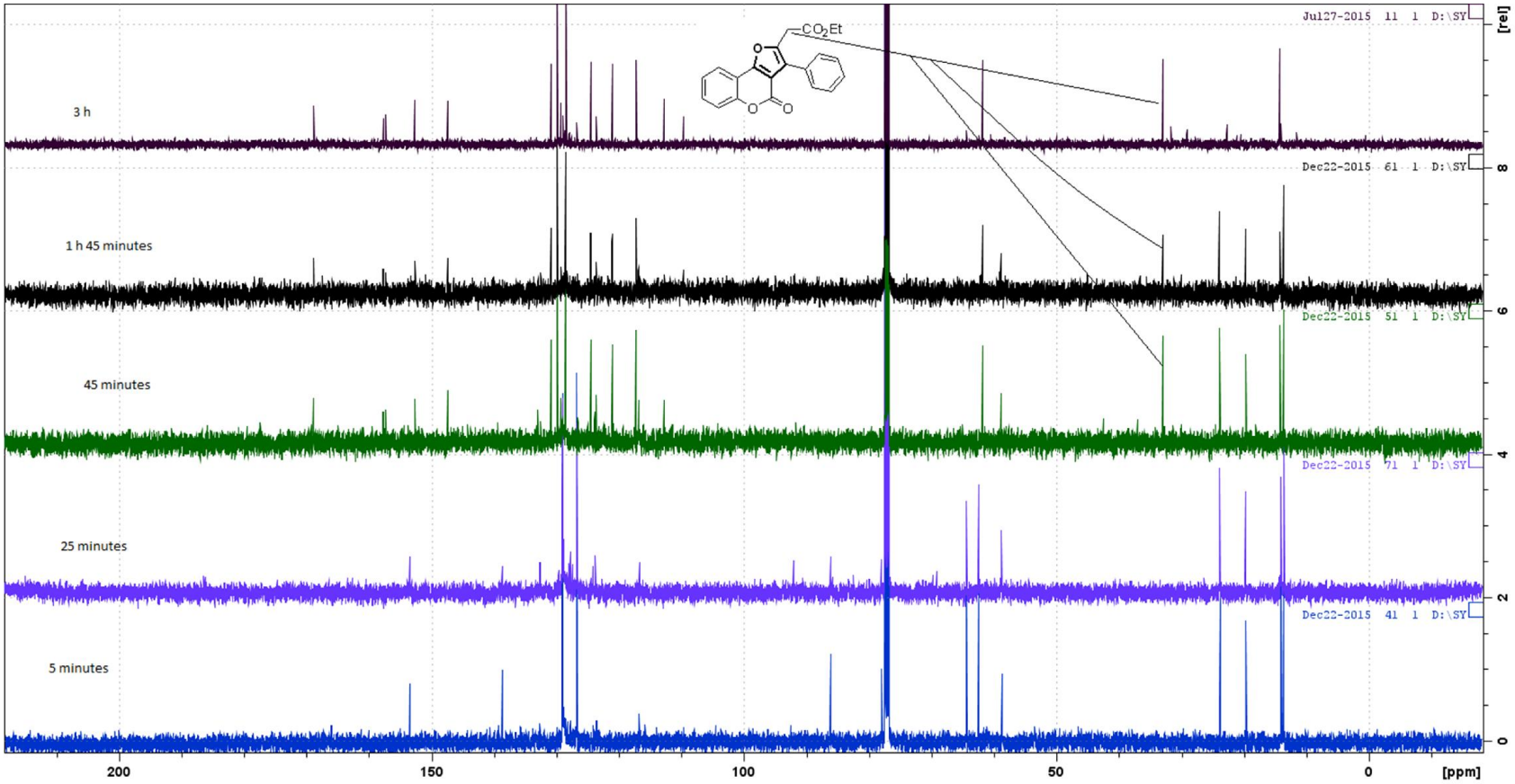

194.20
165.84
148.78
131.73
129.82
127.89
127.09
119.69
119.18
77.34
77.08
76.83
38.69
23.72
22.56
12.05
BRUKER
Me
O
O
10a
Current Data Parameters
NAME Mar04-2016
EXPNO 12
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160305
Time 0.07
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 67
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010040 sec
RG 1030
DW 16.800 usec
DE 6.50 usec
TE 287.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
SFO1 125.8131145 MHz
NUC1 13C
P1 9.70 usec
PLW1 73.00000000 W
CHANNEL f2
SFO2 500.3020012 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 15.13599968 W
PLW12 0.53924000 W
PLW13 0.36512001 W
F2 - Processing parameters
SI 32768
SF 125.8005351 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

SYRK-2-7A


7.412
7.404
7.337
7.328
7.288
2.929
2.917
2.904
2.531
2.519
2.505
2.340
2.219
2.206
BRUKER
Current Data Parameters
NAME Mar05-2016
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160305
Time 14.57
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 128
DW 50.000 usec
DE 6.50 usec
TE 287.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.3030896 MHz
NUC1 1H
P1 15.10 usec
PLW1 15.13599968 W
F2 - Processing parameters
SI 65536
SF 500.3000000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
Me
O
10a
10
9
8
7
6
5
4
3
2
1
0
ppm
3.79
1.18
2.00
2.02
2.91
2.05

SYRK-2-7A


194.19
165.83
148.78
131.73
129.82
127.88
127.08
119.69
119.18
77.33
77.08
76.82
38.69
23.72
22.55
12.05
O
O
Me
10a
BRUKER
Current Data Parameters
NAME Mar05-2016
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160305
Time 15.01
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 32
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 1030
DW 16.800 usec
DE 6.50 usec
TE 288.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.8131145 MHz
NUC1 13C
P1 9.70 usec
PLW1 73.00000000 W
======== CHANNEL f2 ========
SFO2 500.3020012 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 15.13599968 W
PLW12 0.53924000 W
PLW13 0.34512001 W
F2 - Processing parameters
SI 32768
SF 125.8005351 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm



The reaction progress between propargylic alcohol **1a** and 4-hydroxy coumarin **2** was monitored by ^{13}C NMR spectra. The spectra were recorded in five time intervals (5 min to 3 h). In the initial reaction mixture we could notice the presence of ester carbonyl and alkyne carbons at 153.4, 86.1 and 80 ppm respectively. After 45 min, alkyne carbons were completely absent and the ester carbonyl appeared in the downfield at 168.8 ppm because now it is no more a conjugate ester. This is further evidenced by the presence of a methylene (sp^3) carbon at 32.94 ppm which is α to the ester.