Supporting Information

A Mild, Catalytic and Efficient Nazarov Cyclization Medited by Phosphomolybdic Acid

Kaliyappan Murugan, Sankareswaran Srimurugan and Chinpiao Chen*

Department of Chemistry, National Dong Hwa University, Shoufeng, Hualien 974, Taiwan, Republic of China

E-mail: chinpiao@mail.ndhu.edu.tw

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General Methods

Reactions were carried out in flame-dried glassware under a positive nitrogen atmosphere unless otherwise stated. Transfer of anhydrous solvents and reagents was accomplished with oven-dried syringes or cannula. Solvents were distilled before use: methylene chloride, benzene and toluene from calcium hydride, tetrahydrofuran and diethylether from sodium/benzophenone ketyl. Thin layer chromatography was performed on glass plates precoated with 0.25 mm Kieselgel 60 F254 (Merck). Flash chromatography column were packed with 230-400 mesh silica gel. Proton nuclear magnetic resonance spectra (^{1}H NMR) were recorded at 400 MHz, and coupling constants (*J*) are reported in Hertz (Hz). The chemical shifts are reported on the δ scale (ppm) and the spectra are referenced to tetramethylsilane (0 ppm, ^{1}H; ^{13}C) or to deuteriochloroform (7.26 ppm, ^{1}H; 77 ppm, ^{13}C) as internal standard. Carbon nuclear magnetic resonance spectra (^{13}C NMR) were recorded at or 100.6 MHz. Infrared (IR) spectra were measured with a Mattson Galaxy Series FT-IR 3000 spectrophotometer. Mass spectra were determined on a PerSeptive Biosystem Mariner high-resolution electrospray spectrometer in the positive mode.

General Procedures

Activation of Phosphomolybdic acid:

Phosphomolybdic acid hydrate (20%) taken in a single neck round bottom flask which was heated to 110 ºC under reduced pressure for 6h , fine yellow solid obtained which is used for cyclization.

Preparation of PMA/SiO_2 (10% w/w) Catalyst[1] :

To a solution of $H_3PMo_{12}O_{40}$ (100 mg, 0.1 equiv by wt) in MeOH (5 mL) was added slowly silica gel (900 mg, 0.9 equiv by wt, 70-230 mesh), and the mixture was stirred at room temperature for 6 h. Evaporation of MeOH under reduced pressure gave a dry yellowish powder, which contained 10% w/w of PMA.

Preparation of β-keto ester:

Method: A

To a solution of corresponding acid (1 equiv.) in ethanol (10 tims) was added concentrated sulfuric acid (0.1 equiv.).Reaction mixture was heated to refluxed for 2h upon when TLC found complete consumption of the acid. The reaction was allowed to cool down to room temperature and ethanol was removed *in vacuo.* The residue was diluted with ethyl acetate and pH adjusted to 8 to 9 using saturated sodium carbonate solution. The organic phase was separated and the aqueous phase was extracted with ethyl acetate. The combined organic phase was dried over $MgSO_4$, concentrated under

reduced pressure. The residue was purified by flash chromatography (1: 4 EtOAc/hexanes) to afford the ester.

To a suspension of potassium *tert*-butoxide (1.5 equiv.) in ethyl acetate (3 equiv.) was added corresponding ester (1 equiv.). The reaction mixture was heated to reflux for overnight. After completion of the reaction, as indicated by TLC, the reaction was cooled to room temperature and diluted with water, extracted using ethyl acetate , combined ethyl acetate layer dried over $MgSO_4$, concentrated under reduced pressure. The crude product was purified by flash chromatography (1:9, EtOAc/hexanes) to offer the desired β-ketoester as liquid.

Method: B

This method followed the reported procedure[2].

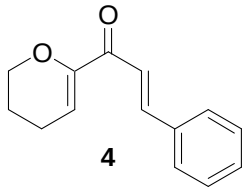
General procedure 1: formation of divnyl alcohol:

This method followed the reported procedure[3].

General procedure 2: Oxidation by MnO_2:

To a suspension of manganese (IV) oxide (7.6 g) in benzene (50 mL) was added a solution of divinyl alcohol (1.0 g, 4.62 mmol) in benzene (50 mL). The reaction was stirred upon when TLC found complete consumption of divnyl alcohol, and then reaction mixture was filtered through celite and washed with EtOAc (4 X 50 mL). The combined filtrate was concentrate *in vacuo.* The product was purified by column chromatography (EtOAc: hexanes = 1:9) to afford 0.800 g (88%) of **4** as yellow solid

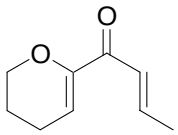
1-(5,6-Dihydro-4*H*-pyran-2-yl)-3-phenylprop-2-en-1-one (4):



4

Yellow solid; 88% yield; Mp 77 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.77 (d, 1H, *J* = 15.8 Hz), 7.58-7.61 (m, 2H), 7.34-7.39 (m, 4H), 6.11 (t, 1H, *J* = 4.3 Hz), 4.15 (t, 2H, *J* = 5.1 Hz), 2.24-2.28 (m, 2H), 1.86-1.90 (m, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ: 185.5, 151.9, 143.8, 135.1, 130.4, 128.9, 128.5, 120.5, 110.8, 66.4, 21.6, 21.0; IR (KBr): 3061, 3025, 2954, 2860, 1655, 1600, 1494, 1441, 1330, 1279, 1179 cm $^{-1}$; MS *m/z*: 214 (M^+, 100), 186 (88), 185 (53), 171 (27), 129 (42), 115 (48), 77 (14); HRMS-EI (*m/z*): $[M]^+$ calcd for $C_{14}H_{14}O_2$, 214.0994; found, 214.0986.

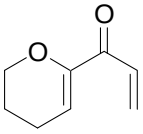
1-(5,6-Dihydro-4*H*-pyran-2-yl)but-2-en-1-one (5):



5

Followed the general procedure[2] afforded **5** as lightly yellow oil; 86% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 6.97-7.06 (m, 1H,), 6.70 (dd, 1H, *J* = 13.6, 1.6 Hz), 6.01 (t, 1H, *J* = 4.2 Hz), 4.11 (t, 2H, *J* = 5.1 Hz), 2.21-2.25 (m, 2H), 1.92 (d, 3H, *J* = 1.6 Hz), 1.85-1.88 (m, 2H); ^{13} C NMR (100.6 MHz, $CDCl_3$) δ: 185.4, 151.6, 143.8, 125.7, 110.7, 66.2, 21.5, 20.8, 18.4; IR ν: 3447, 2942, 1727, 1622, 1443, 1281, 1160 cm^{-1}; MS *m/z*: 153 (M^++1, 21), 137 (100), 124 (26), 109 (21), 81 (38), 67 (18), 53 (25); HRMS-EI (*m/z*): $[M]^+$ calcd for $C_9H_{12}O_2$, 152.0837; found, 152.0833.

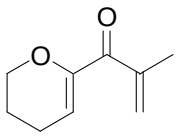
1-(5,6-Dihydro-4*H*-pyran-2-yl)prop-2-en-1-one (6):



6

Followed the general procedure[2] afforded **6** as colorless oil; 75% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 6.87 (q, 1H, *J* = 10.5 Hz), 6.31 (dd, 1H, *J* = 17.1, 1.8 Hz), 6.00 (t, 1H, *J* = 4.2 Hz), 5.68 (dd, 1H, *J* = 10.5, 1.8 Hz), 4.05 (t, 2H, *J* = 5.2 Hz), 2.16-2.21 (m, 2H,), 1.81 (qt, 2H, *J* = 6.2 Hz); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ: 185.6, 151.5, 130.6, 129.1, 112.3, 66.4, 21.4, 20.9; IR ν: 3447, 2934, 1718, 1626, 1400, 1062 cm^{-1}; MS *m/z*: 138 (100), 110 (45), 96 (20), 95 (30), 82 (35), 67 (47), 54 (57), 53 (23); HRMS-EI (*m/z*): $[M]^+$ calcd for $C_8H_{10}O_2$, 138.0681; found, 138.0319.

1-(5,6-Dihydro-4*H*-pyran-2-yl)-2-methylprop-2-en-1-one (7):

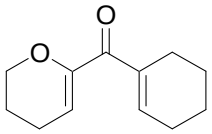


7

Followed the general procedure[2] afforded **7** as colorless oil; 84% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 5.79 (t, 1H, *J* = 4.1 Hz), 5.60 (t, 1H, *J* = 1.0 Hz), 5.57-5.58 (m, 1H), 4.05 (t, 2H, *J* = 5.1 Hz), 2.14-2.18 (m, 2H), 1.87 (t, 3H, *J* = 1.0 Hz), 1.78-1.82 (m, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ: 192.6, 150.8, 142.7, 123.8, 114.1, 66.3, 21.4, 20.8, 18.9; IR ν: 2930, 2877, 1658, 1625, 1446, 1320, 1289, 1234, 1172, 1086 cm^{-1}; MS *m/z*: 152 (M^+, 100), 137 (13), 124 (25), 111 (36), 109 (23), 97 (16), 83 (29), 71

(22), 69 (95), 55 (71); HRMS-EI (*m/z*): $[M]^+$ calcd for $C_9H_{12}O_2$, 152.0837; found, 152.0844.

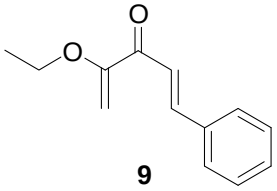
Cyclohexenyl(5,6-dihydro-4*H*-pyran-2-yl)methanone (8):



8

Followed the general procedure[2] afforded **8** as colorless oil; 71% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 6.67-6.70 (m, 1H,), 5.65 (t, 1H, *J* = 4.1 Hz), 4.08 (t, 2H, *J* = 5.2 Hz), 2.16-2.27 (m, 6H,), 1.82-1.88 (m, 2H), 1.57-1.66 (m, 4H,); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ: 192.3, 151.4, 140.3, 137.5, 111.6, 66.3, 25.8, 24.1, 21.9, 21.6, 20.7; IR ν: 2929, 1743 1630, 1435 1276, 1255 cm^{-1}; MS *m/z*: 192 (M^+, 100), 164 (71), 149 (20), 116 (16), 109 (17), 91 (23), 79 (41), 67 (20), 55 (19); HRMS-EI (*m/z*): $[M]^+$ calcd for $C_{12}H_{16}O_2$, 192.1150; found, 192.1147.

4-Ethoxy-1-phenylpenta-1,4-dien-3-one (9):



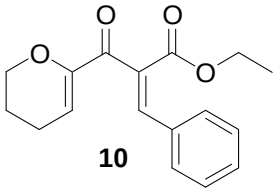
9

Followed the general procedure[2] afforded **9** as Yellow oil; 61% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.76 (d, 1H, *J* = 15.9 Hz), 7.58-7.61 (m, 2H), 7.37-7.39 (m, 4H), 5.30 (d, 1H, *J* = 2.4 Hz), 4.53 (d, 1H, *J* = 2.4 Hz), 3.87 (q, 2H, *J* = 7.0 Hz), 1.43 (t, 3H, *J* = 7.0 Hz); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ: 186.4, 158.2, 144.5, 134.9, 130.5, 128.8, 128.5, 120.6, 91.7, 63.8, 14.3; IR ν: 3080, 3060, 3028, 2981, 1930, 2901, 1736, 1678, 1594cm^{-1}.

General Procedure 3: Knoevenagel Condensation.[4] To 50 mL round bottom flask containing a solution of β-ketoester (380 mg, 1.9 mmol, 1 equiv.) in benzene (11.0 mL) at r.t. was added acetic acid (69 mg, 1.15 mmol, 0.6 equiv.), piperidine (16 mg, 0.19 mmol, 0.1 equiv.) and the proper aldehyde (1.73 mmol, 0.9 equiv.) sequentially. The flask was fitted with a Dean-Stark trap and a condenser. After flushed with argon, this flask was immersed into an oil-bath preheated to 100 ºC. The reaction was refluxed for 1 hour upon when TLC found complete consumption of the β-ketoester. The reaction was allowed to cool down to room temperature and diluted with 5 mL $NaHCO_3$ (aq.). The organic phase was separated and the aqueous phase was extracted

with dichloromethane (5 mL X 3 times). The combined organic phase was dried over $MgSO_4$, concentrated under reduced pressure. The residue was purified by flash chromatography (1:4 EtOAc/hexanes) to afford the desired alkylidene β-ketoester **10** as a colorless liquid.

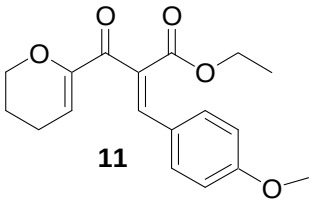
Ethyl 2-(3,4-dihydro-2*H*-pyran-6-carbonyl)-3-phenylacrylate (10):



10

Colorless liquid; 65% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.81 (s, 1H), 7.32-7.38 (m, 5H,), 5.99 (t, 1H, *J* = 4.2 Hz), 4.27 (q, 2H, *J* = 7.1 Hz), 4.07 (t, 2H, *J* = 5.1 Hz), 2.11-2.16 (m, 2H), 1.80 (q, 2H, *J* = 5.2 Hz), 1.28 (t, 3H, *J* = 7.2 Hz); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ: 190.7, 165.0, 151.3, 143.1, 133.1, 130.6, 130.5, 130.3, 128.9, 116.3, 66.6, 61.6, 21.4, 21.1, 14.3; IR ν: 3061, 2941, 2896, 1699, 1663, 1617, 1449, 1293 cm^{-1}; MS *m/z*: 287 (M^++1, 17), 286 (M^+, 76), 241 (19), 213 (46), 212 (100), 185 (21), 156 (18), 128 (51), 71 (22), 115 (32), 77 (14); HRMS-EI (*m/z*): $[M]^+$ calcd for $C_{17}H_{18}O_4$, 286.1205; found, 286.1197.

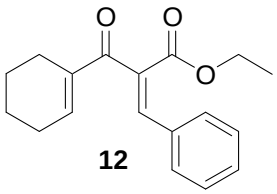
Ethyl 2-(3,4-dihydro-2*H*-pyran-6-carbonyl)-3-(4-methoxyphenyl)acrylate (11):



11

Followed the general procedure 3 afforded **11** as colorless oil; 73% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.75 (s, 1H), 7.34 (d, 2H, *J* = 8.8 Hz), 6.84 (d, 2H, *J* = 8.8 Hz), 6.02 (t, 1H, *J* = 4.2 Hz), 4.25 (q, 2H, *J* = 7.1 Hz), 4.11 (t, 2H, *J* = 5.1 Hz), 3.8 (s, 3H), 2.13-2.22 (m, 2H), 1.80-1.87 (m, 2H), 1.28 (t, 3H, *J* = 7.1 Hz); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ: 191.1, 165.1, 161.3, 151.2, 142.6, 132.3, 127.7, 125.5, 114.2, 66.5, 61.3, 55.3, 21.3, 21.0, 14.2; IR ν: 2935, 1714, 1670, 1625, 1602, 1513, 1250, 1175 cm^{-1}; MS *m/z*: 316 (M^+, 39), 271 (4), 242 (100), 215 (14), 186 (7), 158 (7), 135 (5), 115 (7), 77 (3); HRMS-EI (*m/z*): $[M]^+$ calcd for $C_{18}H_{20}O_5$, 316.1311; found, 316.1303.

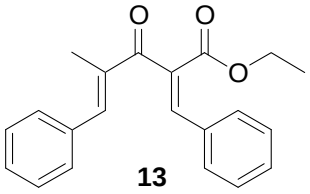
Ethyl 2-(cyclohex-1-enecarbonyl)-3-(4-methoxyphenyl)acrylate (12):



12

Followed the general procedure 3 afforded **12** as lightly yellow oil; 75% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.71 (s, 1H), 7.25-7.29 (m, 2H), 6.80-6.84 (m, 3H), 4.22 (q, 2H, *J* = 7.1 Hz), 3.79 (s, 3H), 2.34-2.37 (m, 2H), 2.12-2.14 (m, 2H), 1.63-1.66 (m, 2H), 1.55-1.58 (m, 2H), 1.25 (t, 3H, *J* = 7.1 Hz); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ: 197.3, 165.5, 161.1, 144.7, 141.2, 139.3, 132.0, 129.0, 125.8, 114.1, 61.1, 55.3, 26.2, 22.7, 21.7, 21.5, 14.2; IR ν: 2936, 2567, 2253, 2050, 1715, 1603, 1513,1463 cm^{-1}; MS *m/z*: 285 (M^++1, 0.5), 284 (M^+, 0.1), 269 (5), 241 (22), 240 (100), 211 (4), 171 (5), 135 (8), 108 (4), 77 (3); HRMS-EI (*m/z*): $[M]^+$ calcd for $C_{18}H_{20}O_3$, 284.1412; found, 284.1417.

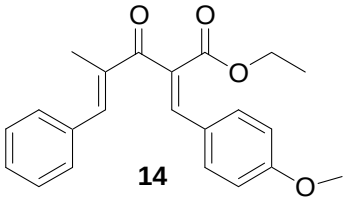
Ethyl 2-benzylidene-4-methyl-3-oxo-5-phenylpent-4-enoate (13):



13

Followed the general procedure 3 afforded **13** as light brownish oil; 97% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.89 (s, 1H), 7.46 (s, 1H), 7.30-7.37 (m, 10H), 4.28 (q, 2H, *J* = 7.2 Hz), 2.19 (s, 3H), 1.28 (t, 3H, *J* = 7.1 Hz; ^{13}C NMR (100.6 MHz, $CDCl_3$) δ: 198.0, 165.2, 143.0, 142.1, 137.0, 135.4, 133.2, 131.6, 130.2, 130.0, 129.9, 129.0, 128.8, 128.4, 61.5, 14.2, 12.6; IR ν: 3058, 3027, 2981, 1715, 1651, 1622, 1448, 1247, 1197 cm^{-1}; MS *m/z*: 320 (M^+, 33), 275 (60), 274 (100), 248 (59), 246 (68), 231 (35), 218 (29), 203 (33), 145 (18), 115 (40), 91 (14), 77 (13); HRMS-EI (*m/z*): $[M]^+$ calcd for $C_{21}H_{20}O_3$, 320.1412; found, 320.1414.

Ethyl 2-(4-methoxybenzylidene)-4-methyl-3-oxo-5-phenylpent-4-enoate (14):

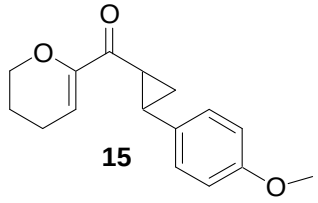


14

Followed the general procedure 3 afforded **14** as Light brownish oil; 92% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.83, (s, 1H), 7.49 (s, 1H), 7.30-7.39 (m, 7H), 6.83 (d, 2H,

J = 8.8 Hz), 4.26 (q, 2H, *J* = 7.1 Hz), 3.79 (s, 3H), 2.22 (d, 3H, *J* = 1.2 Hz), 1.27 (t, 3H, *J* = 7.1 Hz); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ: 198.7, 165.5, 142.8, 141.8, 137.1, 135.5, 132.1, 129.9, 129.0, 128.8, 128.4, 125.7, 114.3, 61.3, 55.3, 14.2, 12.6; IR ν: 2979, 1713, 1653, 1602, 1512, 1305, 1237, 1200, 1176, 1027 cm^{-1}; MS *m/z*: 350 (M^+, 58), 304 (20), 277 (34), 276 (100), 261 (19), 248 (21), 233 (11), 202 (11), 135 (11), 115 (20), 91 (8); HRMS-EI (*m/z*): $[M]^+$ calcd for $C_{22}H_{22}O_4$, 350.1518; found, 350.1512.

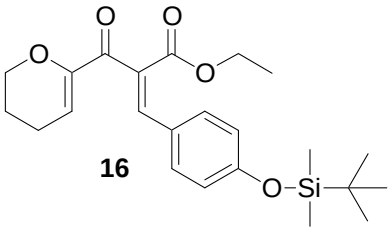
(5,6-Dihydro-4*H*-pyran-2-yl)(2-(4-methoxyphenyl)cyclopropyl)methanone (15):



15

Followed the reported procedure[5]; White solid: Mp 95-96 ºC; 62% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.05 (d, 2H, *J* = 8.6 Hz), 6.82 (d, 2H, *J* = 8.6 Hz), 6.01 (t, 1H, *J* = 4.1 Hz), 4.10 (t, 2H, *J* = 5.0 Hz), 3.78 (s, 3H), 2.62-2.67 (m, 1H), 2.53 (m, 1H), 2.19-2.23 (m, 2H), 1.82-1.88 (m, 2H), 1.68-1.73 (m, 1H), 1.36-1.39 (m, 1H); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ: 194.7, 158.3, 151.4, 132.6, 127.3, 113.9, 109.6, 66.3, 55.3, 29.4, 27.4, 21.5, 20.7, 19.3; IR ν: 2954, 2886, 2835, 1680, 1665, 1625, 1515, 1441, 1395, 1290, 1235, 1060 cm^{-1}; MS *m/z*: 258 (M^+, 31), 176 (15), 147 (100), 134 (12), 121 (23), 117 (25), 91 (17), 77 (8); HRMS-EI (*m/z*): $[M]^+$ calcd for $C_{16}H_{18}O_3$, 258.1256; found, 258.1245.

Ethyl 3-(4-(*tert*-butyldimethylsilyloxy)phenyl)-2-(3,4-dihydro-2*H*-pyran-6-carbonyl)acrylate (16):

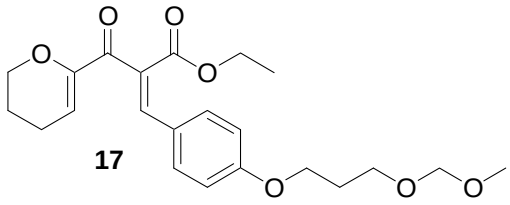


16

Followed the general procedure 3 afforded **15** as Brown oil; 30% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.74 (s, 1H), 7.27 (d, 2H, *J* = 8.7 Hz), 6.77 (d, 2H, *J* = 8.6 Hz), 6.01 (t, 1H, *J* = 4.2 Hz), 4.25 (q, 2H, *J* = 7.1 Hz) 4.09-4.12 (m, 2H), 2,12-2,17 (m, 2H), 1.77-1.85 (m, 2H), 1.27 (t, 3H, *J* = 7.1 Hz), 0.95 (s, 9H), 0.19 (s, 6H); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ: 191.1, 165.2, 157.9, 151.2, 142.8, 132.2, 127.9, 126.1, 120.3, 116.2, 66.5, 61.3, 25.5, 21.3, 21.0, 18.2, 14.2, –4.3; IR ν: 2955, 2931, 2859, 1717, 1674, 1625, 1599, 1509, 1255, 1173, 913 cm^{-1}; MS *m/z*: 417 (M^++1, 20), 416 (M^+,

68), 371 (4), 343 (31), 342 (100), 285 (20), 143 (8), 73 (13); HRMS-EI (*m/z*): $[M]^+$ calcd for $C_{23}H_{32}O_5Si$, 416.2019; found, 416.2023.

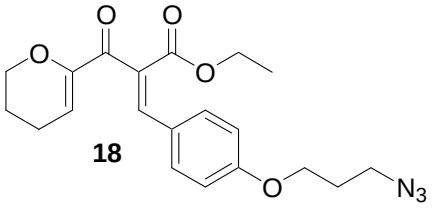
Ethyl 2-(3,4-dihydro-2*H*-pyran-6-carbonyl)-3-(4-(3-(methoxymethoxy)propoxy) phenyl)acrylate (17):



17

Followed the general procedure 3 afforded **17** as Yellow oil; 70% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.73 (s, 1H), 7.30-7.33 (m, 2H), 6.81-6.84(m, 2H), 6.01 (t, 1H, *J* = 4.2 Hz), 4.60 (d, 2H, *J* = 3.0 Hz), 4.24 (q, 2H, *J* = 7.0 Hz), 4.05-4.10 (m, 4H), 3.68 (t, 2H, *J* = 6.1 Hz), 3.32 (s, 3H), 2.12-2.16 (m, 2H), 2.02-2.08 (m, 2H), 1.79-1.84 (m, 2H), 1.26 (t, 3H, *J* = 7.1 Hz); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ: 191.1, 165.2, 160.8, 151.3, 142.7, 132.3, 127.7, 125.5, 116.0, 114.7, 96.4, 66.5, 64.8, 63.9, 61.3, 55.2, 29.4, 21.3, 21.0, 14.2; IR ν: 3516, 2987, 1711, 1602, 1513, 1469, 1367, 1204 cm^{-1}; MS *m/z*: 405 (M^++1, 18), 404 (M^+, 78), 359 (30), 330 (100), 268 (35), 227 (11), 128 (9), 115 (13), 103 (18), 73 (9), 46 (8); HRMS-EI (*m/z*): $[M]^+$ calcd for $C_{22}H_{28}O_7$, 404.1835; found, 404.1841.

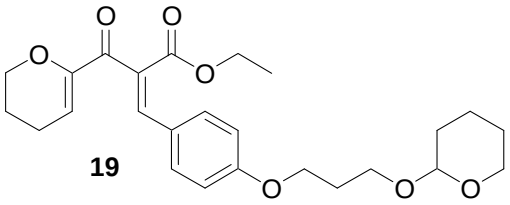
Ethyl 3-(4-(3-azidopropoxy)phenyl)-2-(3,4-dihydro-2*H*-pyran-6-carbonyl) Acrylate (18):



18

Followed the general procedure 3 afforded **19** as yellow oil; 70% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.74 (s, 1H), 7.33 (d, 2H, *J* = 8.7 Hz), 6.83 (d, 2H, *J* = 8.8 Hz), 6.01 (t, 1H, *J* = 4.2 Hz), 4.25 (q, 2H, *J* = 7.1 Hz), 4.03-4.12 (m, 4H), 3.50 (t, 2H, *J* = 6.5 Hz), 2.12-2.17 (m, 2H), 2.01-2.07 (m, 2H), 1.79-1.85 (m, 2H), 1.27 (t, 3H, *J* = 7.1 Hz); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ: 191.0, 165.2, 160.4, 151.3, 142.5, 132.3, 128.0, 125.8, 116.0, 114.7, 66.5, 64.5, 61.3, 48.0, 28.6, 21.3, 21.0, 14.2; IR ν: 3413, 2937, 2098, 1717, 1625, 1602, 1511, 1301, 1250, 1053 cm^{-1}; MS *m/z*: 386 (M^++1, 22), 385 (M^+, 100), 356 (12), 340 (13), 311 (53), 302 (23), 228 (16), 147 (23), 121 (25), 83 (17), 56 (86), 55 (39); HRMS-EI (*m/z*): $[M]^+$ calcd for $C_{20}H_{23}N_3O_5$, 385.1638; found, 385.1643.

Ethyl 2-(3,4-dihydro-2*H*-pyran-6-carbonyl)-3-(4-(3-(tetrahydro-2*H*-pyran-2-yloxy)propoxy)phenyl)acrylate (19):

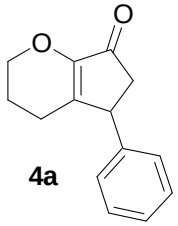


19

Followed the general procedure 3 afforded **19** as Colorless oil; 75% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.73 (s, 1H), 7.32 (d, 1H, *J* = 8.8 Hz), 6.83 (d, 2H, *J* = 8.8 Hz), 6.01 (t, 1H, *J* = 4.2 Hz), 4.56-4.58 (m, 1H), 4.25 (q, 2H, *J* = 7.1 Hz), 4.06-4.11 (m, 4H), 3.81-3.91 (m, 2H), 3.49-3.56 (m, 2H), 2.12-2.15 (m, 2H), 2.03-2.07 (m, 2H), 1.69-1.83 (m, 4H), 1.49-1.57 (m, 4H), 1.27, (t, 3H, *J* = 7.1 Hz; ^{13}C NMR (100.6 MHz, $CDCl_3$) δ: 191.1, 165.2, 160.8, 151.3, 142.7, 132.3, 127.6, 125.4, 116.0, 114.7, 98.9, 66.5, 65.0, 63.8, 62.3, 61.2, 30.6, 29.5, 25.4, 21.3, 21.0, 19.6, 14.2; IR ν: 3421, 2940, 1716, 1625, 1601, 1511, 1467, 1368, 1247, 1175 cm^{-1}; MS *m/z*: 444 (M^+, 8), 360 (30), 386 (45), 228 (7), 143 (22), 85 (100), 84 (56), 83 (34), 57 (32), 56 (36), 55 (46); HRMS-EI (*m/z*): $[M]^+$ calcd for $C_{25}H_{32}O_7$, 444.2148; found, 444.2153.

General Procedure 4: Nazarov Cyclization using Phosphomolybdic acid: To a solution of divinyl ketone **4a** (100 mg, 0.466 mmol, 1.0 equiv.) in acetonitrile (3 mL) was added PMA (4.2 mg, 0.0023 mmol, 0.005 equiv.). The reaction was stirred at 40 ºC. After completion of the reaction, as indicated by TLC, the reaction mixture was filtered. The filtrate was concentrated under reduced pressure, and the crude compound was purified by flash chromatography (1: 2.3, EtOAc/hexanes) to afford the desired Nazarov product **4a**.

5-Phenyl-3,4,5,6-tetrahydrocyclopenta[*b*]pyran-7(2*H*)-one (4a):

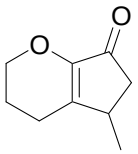


4a

Yellow solid; Mp 86 ºC; 99% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.25-7.31 (m, 2H,), 7.19-7.23 (m, 1H), 7.08-7.10 (m, 2H,), 4.05-4.10 (m, 2H,), 3.81 (dd, 2H, *J* = 6.5, 1.3 Hz), 2.86 (dd, 1H, *J* = 18.9, 6.6 Hz), 2.25 (d, 1H, *J* = 19.0 Hz), 2.09 (dq, 2H, *J* = 18.5, 6.1 Hz), 1.88-1.92 (m, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ: 200.0, 151.5, 147.6, 141.7, 129.0, 127.2, 127.1, 67.0, 43.6, 42.9, 22.2, 21.4; IR ν: 2925, 2866, 1708,

1650, 1454, 1395, 1297, 1116, 1071 cm^{-1}; MS *m/z*: 214 (M^+, 89), 213 (52), 186 (18), 131 (100), 103 (67), 77 (40); HRMS-EI (*m/z*): $[M]^+$ calcd for $C_{14}H_{14}O_2$, 214.0994; found, 214.0987.

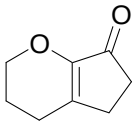
5-Methyl-3,4,5,6-tetrahydrocyclopenta[*b*]pyran-7(2*H*)-one (5a):



5a

Followed the general procedure 4 afforded **5a** lightly yellow oil; 96% yield; ^1H NMR (400 MHz, $CDCl_3$) δ: 4.09-4.14 (m, 1H), 4.01-4.06 (m, 1H), 2.73 (m, 1H), 2.59 (dd, 1H, *J* = 18.6, 6.1 Hz), 2.438-2.47 (m, 1H), 2.16-2.24 (m, 1H,), 1.91-1.97 (m, 3H), 1.15 (d, 3H, *J* = 7.0 Hz) ^{13}C NMR (100.6 MHz, $CDCl_3$) δ: 200.0, 150.4, 149.5, 66.6, 41.5, 32.0, 21.8, 21.4, 19.2; IR ν: 3398, 2959, 2873, 1703, 1645, 1409, 1288, 1120, 1080 cm^{-1}; MS *m/z*: 152 (M^+, 1), 145 (4), 117 (70), 99 (24), 71 (69), 69 (100), 57 (18), 55 (15); HRMS-EI (*m/z*): $[M]^+$ calcd for $C_9H_{12}O_2$, 152.0837; found, 152.0832.

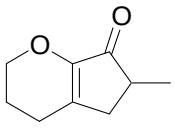
3,4,5,6-Tetrahydrocyclopenta[*b*]pyran-7(2*H*)-one (6a):



6a

Followed the general procedure 4 afforded **6a** colorless oil; 90% yield; ^1H NMR (400 MHz, $CDCl_3$) δ: 4.10 (t, 2H, *J* = 5.2 Hz), 2.45 (t, 2H, *J* = 2.2 Hz), 2.34-2.39 (m, 4H), 1.92-1.98 (m, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ: 200.9, 151.4, 145.7, 67.0, 32.7, 25.8, 24.3, 21.7; IR ν: 3401, 2926, 1700, 1650, 1401, 1291, 1168, 1114, 1073 cm^{-1}; MS *m/z*: 138 (M^+, 16), 114 (20), 105 (29), 83 (19), 77 (22), 69 (69), 57 (100), 55 (92); HRMS-EI (*m/z*): $[M]^+$ calcd for $C_8H_{10}O_2$, 138.0681; found, 138.0678.

6-Methyl-3,4,5,6-tetrahydrocyclopenta[*b*]pyran-7(2*H*)-one (7a):

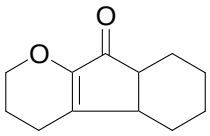


7a

Followed the general procedure 4 afforded **7a** colorless oil; 90% yield; ^1H NMR (400 MHz, $CDCl_3$) δ: 4.05-4.08 (m, 2H,), 2.63-2.70 (m, 1H,), 2.33-2.37 (m, 1H,), 2.30 (t, 2H, *J* = 6.2 Hz), 2.03 (dd, 1H, *J* = 17.4, 1.7 Hz), 1.89-1.93 (m, 2H,), 1.16 (d, 3H, 7.4 Hz); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ: 203.5, 150.1, 143.9, 66.7, 38.0, 34.8, 24.0,

21.6, 16.5; IR ν: 2929, 1705, 1648, 1444, 1402, 1294 cm^{-1}; MS *m/z*: 152 (M^+, 96), 137 (44), 116 (100), 109 (18), 95 (27), 88 (25), 68 (36), 67 (42), 55 (28), 53 (21); HRMS-EI (*m/z*): $[M]^+$ calcd for $C_9H_{12}O_2$, 152.0837; found, 152.0842.

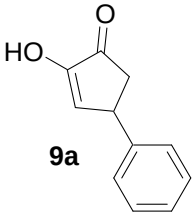
3,4,4b,5,6,7,8,8a-Octahydroindeno[2,1-*b*]pyran-9(2*H*)-one (8a):



8a

Followed the general procedure 4 afforded **8a** colorless oil; 90% yield; ^1H NMR (400 MHz, $CDCl_3$) δ: 4.09-4.12 (m, 1H), 4.04-4.07 (m, 1H), 2.72 (q, 1H, *J* = 8.2 Hz), 2.32-2.43 (m, 2H,), 2.22 (dt, 1H, 18.6, 5.7 Hz), 1.90-1.96 (m, 3H), 1.74-1.80 (m, 1H), 1.69-1.72 (m, 1H), 1.46-1.50 (m, 2H), 1.32-1.36 (m, 2H), 1.17-1.19 (m, 1H); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ: 202.8, 150.3, 148.2, 66.8, 43.7, 37.5, 26.7, 22.4, 22.0, 21.6, 20.5, 20.4; IR ν: 2931, 2863, 1708, 1645, 1290, 1081 cm^{-1}; MS *m/z*: 192 (M^+, 14), 167 (16), 117 (24), 109 (100), 81 (54), 71 (29), 53 (18); HRMS-EI (*m/z*): $[M]^+$ calcd for $C_{12}H_{16}O_2$, 192.1150; found, 192.1143.

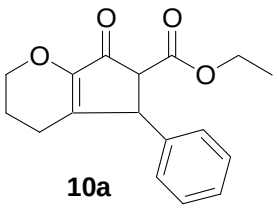
4-Phenylcyclopent-2-enone (9a):



9a

Followed the general procedure 4 afforded **9a** yellow oil; 50% yield; ^1H NMR (400 MHz, $CDCl_3$) δ: 7.32 (t, 2H, *J* = 7.6 Hz), 7.23-7.27 (m, 1H), 7.18 (d, 2H, *J* = 7.0 Hz), 6.58 (d, 1H, *J* = 3.0 Hz), 5.64 (s, 1H), 4.02-4.05 (m, 1H), 2.98 (dd, 1H, *J* = 6.4, 19.4 Hz), 2.34 (dd, 1H, *J* = 1.8, 19.4 Hz); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ: 203.4, 152.6, 142.5, 131.8, 128.9, 127.2, 127.0, 42.1, 39.9; IR ν: 3336, 3082, 3064, 3026, 2920, 2850, 1709, 1650, 1396, 765, 700, 709 cm^{-1}.

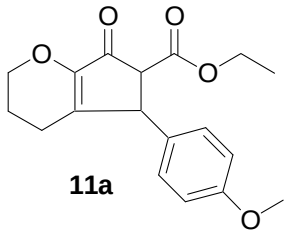
Ethyl 7-oxo-5-phenyl-2,3,4,5,6,7-hexahydrocyclopenta[*b*]pyran-6-carboxylate (10a):



10a

Followed the general procedure 4 afforded **10a** White solid; Mp 125-126 ºC; 99% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.28-7.36 (m, 3H), 7.13 (d, 2H, *J* = 6.8 Hz), 4.12-4.24 (m, 5H), 3.30 (s, 1H), 2.18-2.24 (m, 1H,), 2.00-2.13 (m, 1H,), 1.89-1.99(m, 2H), 1.28 (t, 3H, *J* = 7.1 Hz); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ:193.1, 168.3, 149.8, 147.4, 139.8, 129.1, 127.6, 127.3, 67.0, 61.8, 59.3, 47.6, 22.2, 21.2, 14.1; IR ν: 2979, 2938, 2346, 1735, 1649, 1459, 1326, 1247, 1171. 1051 cm^{-1}; MS *m/z*: 287 (M^++1, 17), 286 (M^+, 100), 257 (18), 241 (16), 212 (92), 128 (20), 102 (19), 77 (17), 55 (19); HRMS-EI (*m/z*): $[M]^+$ calcd for $C_{17}H_{18}O_4$, 286.1205; found, 286.1197.

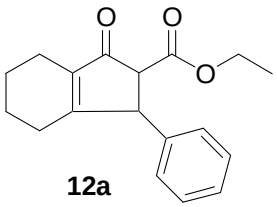
Ethyl 5-(4-methoxyphenyl)-7-oxo-2,3,4,5,6,7-hexahydrocyclopenta[*b*]pyran-6-carboxylate (11a):



11a

Followed the general procedure 4 afforded **11a** lightly yellow oil; 98% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.03 (d, 2H, *J* = 8.5 Hz), 6.84 (d, 2H, *J* = 8.5 Hz), 4.19 (q, 2H, *J* = 7.1 Hz), 4.11-4.14 (m, 3H), 3.77 (s, 3H), 3.24(d, 1H, *J* = 1.6 Hz), 2.14-2.22 (m, 1H), 2.03-2.11 (m, 1H), 1.84-2.01 (m, 2H), 1.25 (t, 3H, *J* = 7.1 Hz); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ: 193.3, 168.4, 159.0, 149.6, 147.8, 131.7, 128.4, 114.5, 67.0, 61.8, 59.5, 55.3, 47.0, 22.2, 21.3,14.1; IR ν: 2935, 2837, 1737, 1649, 1610, 1512, 1249 cm^{-1}; MS *m/z*: 317 (M^++1, 16), 316 (M^+, 90), 287 (12), 271 (11), 243 (25), 242 (100), 233 (35), 214 (17), 160 (25), 137 (11), 55 (16); HRMS-EI (*m/z*): $[M]^+$ calcd for $C_{18}H_{20}O_5$, 316.1311; found, 316.1307.

Ethyl -(4-methoxyphenyl)-3-oxo-2,3,4,5,6,7-hexahydro-1H-indene-2-carboxylate (12a):[1]

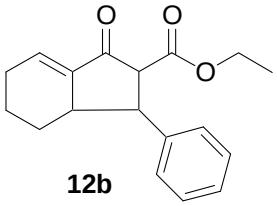


12a

Followed the general procedure 4 afforded **12a** lightly yellow oil; 78% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 6.98-7.01 (m, 2H), 6.83-6.86 (m, 2H), 4.19 (q, 2H, *J* = 4.2 Hz), 3.31 (d, 1H, *J* = 2.8 Hz), 2.21-2.23 (m, 2H), 2.06-2.16 (m, 2H), 1.61-1.71 (m, 5H), 1.27 (t, 3H, *J* = 7.1 Hz); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ: 200.8, 175.6, 169.0, 158.9, 137.4, 132.0, 128.5, 114.4, 61.6, 55.3, 53.4, 51.3, 26.4, 22.0, 21.4, 20.3, 14.2;

IR ν: 2936, 1717, 1687, 1647, 1512, 1463, 1248 cm^{-1}; MS *m/z*: 284 (M^+, 5), 268 (24), 241 (93), 161 (18), 135 (21), 108 (100), 80 (28), 79 (35); HRMS-EI (*m/z*): $[M]^+$ calcd for $C_{18}H_{20}O_3$, 284.1412; found, 284.1424.

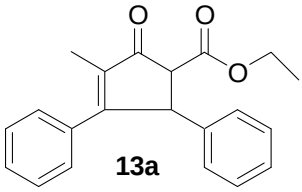
Ethyl 1-(4-methoxyphenyl)-3-oxo-2,3,5,6,7,7a-hexahydro-1*H*-indene-2-carboxylate (12b):



12b

Followed the general procedure 4 afforded **12b** lightly yellow oil; 22% yield; ^1H NMR (400 MHz, $CDCl_3$) δ: 7.20 (d, 2H, *J* = 8.6 Hz), 6.86-6.88 (m, 3H), 4.05-4.21 (m, 2H), 3.79 (s, 3H), 3.46 (d, 1H, *J* = 12.4 Hz), 3.17 (t, 1H, *J* = 12.0 Hz), 2.58-2.63 (m, 1H), 2.31-2.37 (m, 1H), 2.16-2.27 (m, 1H), 1.95-2.01 (m, 1H), 1.85-1.89 (m, 1H), 1.45-1.52 (m, 1H), 1.20 (t, 3H, *J* = 7.1 Hz); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ: 197.7, 169.1, 158.7, 139.5, 135.6, 131.5, 128.2, 114.1, 62.4, 61.3, 55.2, 50.6, 43.2, 26.7, 25.7, 21.5, 14.2; IR ν: 2936, 1739, 1719, 1651, 1514, 1251 cm^{-1}; MS *m/z*: 285 (M^++1, 2), 268 (100), 267 (48), 240 (23), 237 (37), 209 (19), 161 (14), 109 (27), 108 (31), 81 (33), 79 (25), 53 (12); HRMS-EI (*m/z*): $[M]^+$ calcd for $C_{18}H_{20}O_3$, 284.1412; found, 284.1418.

Ethyl 3-methyl-2-oxo-4,5-diphenylcyclopent-3-enecarboxylate (13a):

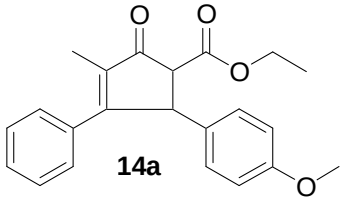


13a

Followed the general procedure 4 afforded **13a** lightly yellow oil; 90% yield; ^1H NMR (400 MHz, $CDCl_3$) δ: 7.08-7.39(m, 10H), 4.92 (s, 1H), 4.24-4.32 (m, 2H), 3.49 (d, 1H, *J* = 2.7 Hz), 2.05 (d, 3H, *J* = 1.4 Hz), 1.33 (t, 3H, *J* = 7.1 Hz); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ: 201.6, 169.0, 168.6, 140.7, 135.9, 134.5, 129.4, 128.9, 128.4, 128.3, 127.6, 127.1, 61.8, 61.7, 50.99 14.2, 10.3; IR ν: 3061, 3028, 2981, 1732, 1700, 1626, 1444, 1341, 1249,1158, 1019 cm^{-1}; MS *m/z*: 321 (M^++1, 12), 320 (M^+, 54), 274 (44), 273 (100), 247 (95), 145 (29), 117 (46), 115 (59), 91 (37), 77 (16); HRMS-EI (*m/z*): $[M]^+$ calcd for $C_{21}H_{20}O_3$, 320.1412; found, 320.1423.

Ethyl 2-(4-methoxyphenyl)-4-methyl-5-oxo-3-phenylcyclopent-3-enecarboxylate

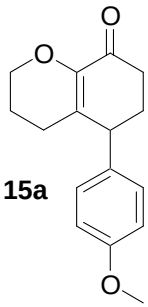
(14a):



14a

Followed the general procedure 4 afforded **14a** light yellowish oil; 88% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.27-7.35 (m, 5H), 6.98 (d, 2H, *J* = 8.6 Hz), 6.72 (d, 2H, *J* = 8.6 Hz), 4.84 (s, 1H), 4.21-4.29 (m, 2H), 3.7 (s, 3H), 3.43 (d, 1H, *J* = 2.7 Hz), 2.01 (d, 3H, *J* = 1.8 Hz) 1.31 (t, 3H, *J* = 7.1 Hz); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ: 201.8, 169.2, 168.7, 158.3, 135.7, 134.5, 132.7, 129.3, 128.6, 128.4, 128.3, 114.2, 61.8, 61.8, 55.1, 53.4, 50.2, 14.2, 10.2; IR ν: 2930, 1732, 1709, 1611, 1512, 1443, 1341, 1248,1177, 1030 cm^{-1}; MS *m/z*: 351 (M^++1, 11), 350 (M^+, 50), 304 (49), 303 (100), 277 (69), 233 (28), 145 (26), 121 (77), 117 (55), 115 (56), 91 (29), 77 (11); HRMS-EI (*m/z*): $[M]^+$ calcd for $C_{22}H_{22}O_4$, 350.1518; found, 350.1524.

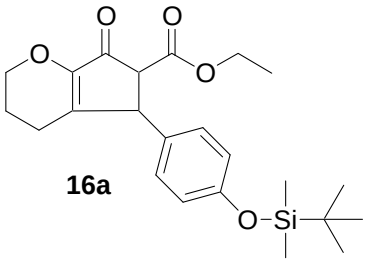
5-(4-Methoxyphenyl)-3,4,6,7-tetrahydro-2*H*-chromen-8(5*H*)-one (15a):



15a

Followed the general procedure 4 afforded **15a** Yellow oil; 80% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.12 (d, 2H, *J* = 8.6 Hz), 6.88 (d, 2H, *J* = 8.6 Hz), 4.05-4.10 (m, 2H), 3.79 (s, 3H), 3.56 t, 1H, *J* = 5.4 Hz), 2.47-2.53 (m, 1H), 2.29-2.42 (m, 2H), 1.93-1.98 (m, 3H), 1.81-1.85 (m, 2H); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ: 193.2, 158.6, 146.8, 133.3, 132.1, 129.0, 114.1, 65.9, 55.3, 45.0, 34.9, 30.9, 25.1, 21.8; IR ν: 3436, 2936, 1673, 1610, 1511, 1464, 1382, 1248, 1153, 1084, 1034 cm^{-1}; MS *m/z*: 259 (M^++1, 14), 258 (M^+, 100), 230 (23), 216 (31), 202 (19), 188 (17), 173 (12), 159 (13), 115 (14), 91 (10), 77 (12); HRMS-EI (*m/z*): $[M]^+$ calcd for $C_{16}H_{18}O_3$, 258.1256; found, 258.1265.

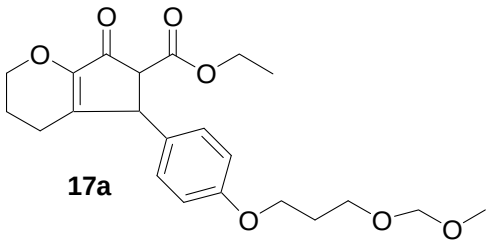
Ethyl 5-(4-(*tert*-butyldimethylsilyloxy)phenyl)-7-oxo-2,3,4,5,6,7-hexahydro cyclopenta[*b*]pyran-6-carboxylat (16a):



16a

Followed the general procedure 4 afforded **16a**. Yellowish oil; 94% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 6.96 (d, H, *J* = 8.4 Hz), 6.78 (d, 2H, *J* = 8.4 Hz), 4.20 (q, 2H, *J* = 7.2 Hz), 4.11-4.18 (m, 3H), 3.25 (d, 1H, 1.7 Hz), 2.15-2.26 (m, 1H), 2.03-2.11 (m, 1H), 1.92-1.98 (m, 2H), 1.27 (t, 3H, *J* = 7.1 Hz), 0.96 (s, 9H), 0.1 (s, 3H); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ: 193.3, 168.5, 155.1, 149.6, 147.8, 132.3, 128.3, 120.6, 67.1, 61.8, 59.5, 47.0, 25.6, 22.2, 21.3, 18.1, 14.2; IR ν: 2955, 2931, 2858, 1737, 1714, 1650, 1607, 1509, 1262, 1123, 914 cm^{-1}; MS *m/z*: 417 (M^++1, 21), 416 (M^+, 70), 359 (26), 342 (16), 313 (17), 285 (16), 129 (12), 75 (100), 55 (17); HRMS-EI (*m/z*): $[M]^+$ calcd for $C_{23}H_{32}O_5Si$, 416.2019; found, 416.2009.

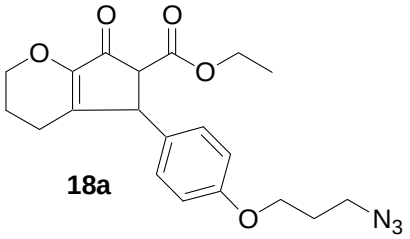
Ethyl 5-(4-(3-(methoxymethoxy)propoxy)phenyl)-7-oxo-2,3,4,5,6,7-hexahydro cyclopenta[*b*]pyran-6-carboxylate (17a):



17a

Followed the general procedure 4 afforded **17a** yellow oil; 80% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.01 (d, 2H, *J* = 8.6 Hz), 6.86 (d, 2H, *J* = 8.6 Hz), 4.62(s, 2H), 4.21 (q, 2H, *J* = 7.1 Hz), 4.12-4.16 (m, 3H), 4.05 (t, 2H, *J* = 6.2 Hz), 6.71 (t, 2H, *J* = 6.1 Hz), 3.34 (s, 3H), 3.25 (d, 1H, *J* = 2.3 Hz), 2.16-2.22 (m, 1H), 2.02-2.12 (m, 3H), 1.89-1.96 (m, 2H), 1.27 (t, 3H, *J* = 7.1 Hz); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ: 193.2, 168.4, 158.4, 149.7, 147.6, 131.7, 128.3, 115.1, 96.5, 67.0, 64.8, 64.1, 61.8, 59.5, 55.2, 47.0, 29.6, 22.2, 21.3, 14.1; IR ν: 3436, 2935, 1717, 1651, 1611, 1511, 1368, 1247, 1123, 1043 cm^{-1}; MS *m/z*: 405 (M^++1, 24), 404 (M^+, 100), 359 (27), 330 (46), 268 (24), 103 (62), 73 (23), 71 (37), 55 (22); HRMS-EI (*m/z*): $[M]^+$ calcd for $C_{22}H_{28}O_7$, 404.1835; found, 404.1833.

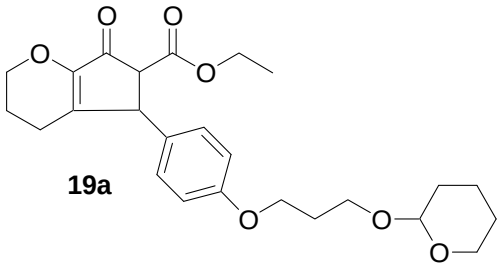
Ethyl 5-(4-(3-azidopropoxy)phenyl)-7-oxo-2,3,4,5,6,7-hexahydrocyclopenta[*b*] pyran-6-carboxylate (18a):



18a

Followed the general procedure 4 afforded **18a** Yellow oil; 85% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.03-7.05 (m, 2H), 6.85-6.87 (m, 2H), 4.21 (q, 2H, *J* = 7.0 Hz), 4.12-4.16 (m, 3H), 4.03 (t, 2H, *J* = 5.9 Hz), 3.51 (t, 2H, *J* = 6.6 Hz), 3.24 (d, 1H, *J* = 2.3 Hz), 1.91-2.18 (m, 7H), 1.27 (t, 3H, *J* = 7.1 Hz); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ: 193.2, 168.4, 158.1, 149.7, 147.5, 132.1, 128.4, 115.1, 67.0, 64.5, 61.8, 59.5, 48.2, 47.0, 28.7, 22.2, 21.3, 14.1; IR ν: 3426, 2938, 2098, 1720, 1651, 1511, 1368, 1298, 1246, 1123 cm^{-1}; MS *m/z*: 386 (M^++1, 19), 385 (M^+, 93), 311 (100), 228 (23), 201 (16), 115 (16), 56 (36); HRMS-EI (*m/z*): $[M]^+$ calcd for $C_{20}H_{23}N_3O_5$, 385.1638; found, 385.1630.

Ethyl 7-oxo-5-(4-(3-(tetrahydro-2*H*-pyran-2-yloxy)propoxy)phenyl)-2,3,4,5,6,7-hexahydrocyclopenta[*b*]pyran-6-carboxylate (19a):



19a

Followed the general procedure 4 afforded **19a** lightly yellow oil; 75% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.02 (d, 2H, *J* = 8.6 Hz), 6.86 (d, 2H, *J* = 8.6 Hz), 4.58 (t, 1H, *J* = 4.4 Hz), 4.22 (q, 2H, *J* = 7.0 Hz), 4.12-4.18 (m, 3H), 4.06 (dt, 2H, *J* = 2.1, 6.3 Hz), 3.88-3.93 (m, 1H), 3.80-3.86 (m, 1H), 3.51-3.57 (m, 1H), 3.46-3.50 (m, 1H), 3.25 (d, 1H, *J* = 2.3 Hz), 2.16-2.17 (m, 1H), 2.03-2.11 (m, 3H), 1.89-1.97 (m, 2H), 1.79-1.82 (m, 1H), 1.67-1.70 (m, 1H), 1.49-1.59 (m, 4H), 1.27 (t, 3H, *J* = 7.2 Hz); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ: 193.3, 168.4, 158.5, 149.6, 147.7, 131.6, 128.3, 115.1, 98.9, 67.0, 65.0, 63.9, 62.3, 61.8, 59.5, 47.0, 30.6, 29.6, 25.4, 22.2, 21.3, 19.6, 14.1; IR ν: 2935, 1717, 1649, 1610, 1511, 1247, 1123, 1034 cm^{-1}; MS *m/z*: 445 (M^++1, 7), 444 (M^+, 24), 360 (71), 332 (34), 314 (22), 286 (64), 277 (252), 232 (27), 204 (51), 173 (23), 147 (35), 146 (67), 143 (61), 121 (33), 87 (41), 85 (89), 84 (100), 83 (60), 71 (72), 55 (92); HRMS-EI (*m/z*): $[M]^+$ calcd for $C_{25}H_{32}O_7$, 444.2148; found, 444.2140.

References

1. G. D. Kishore kumar, S. Baskaran, *J. Org. Chem.*, 2005, **70**, 4520–4523.
2. K. A. Varinder, J. B. Andrew, *Org. Lett.*, 2003, **5**, 5075–5078.
3. L. Guangxin, N. G. Stefan, D. Trauner, *Org. Lett.*, 2003, **5**, 4931–4934.
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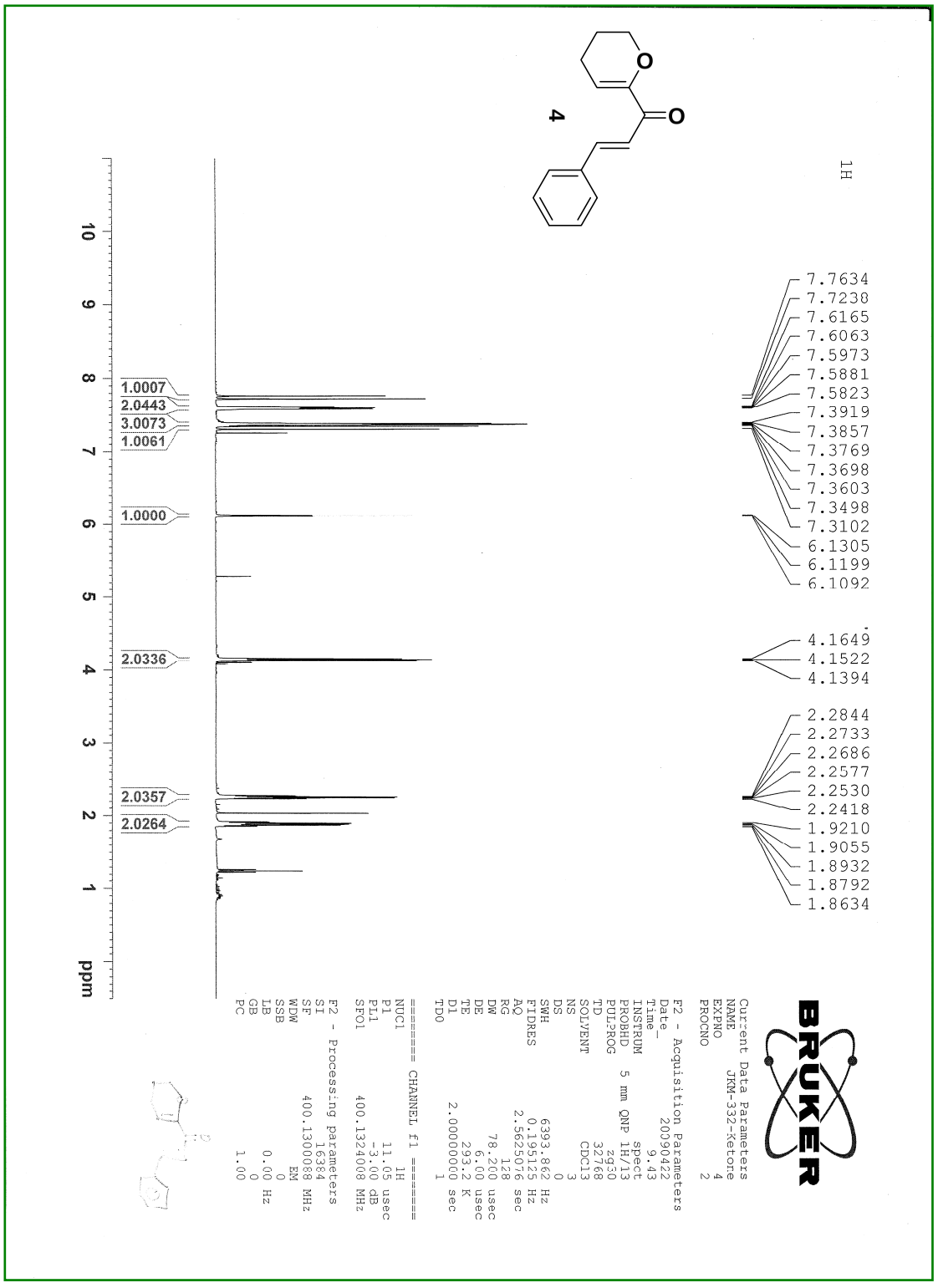
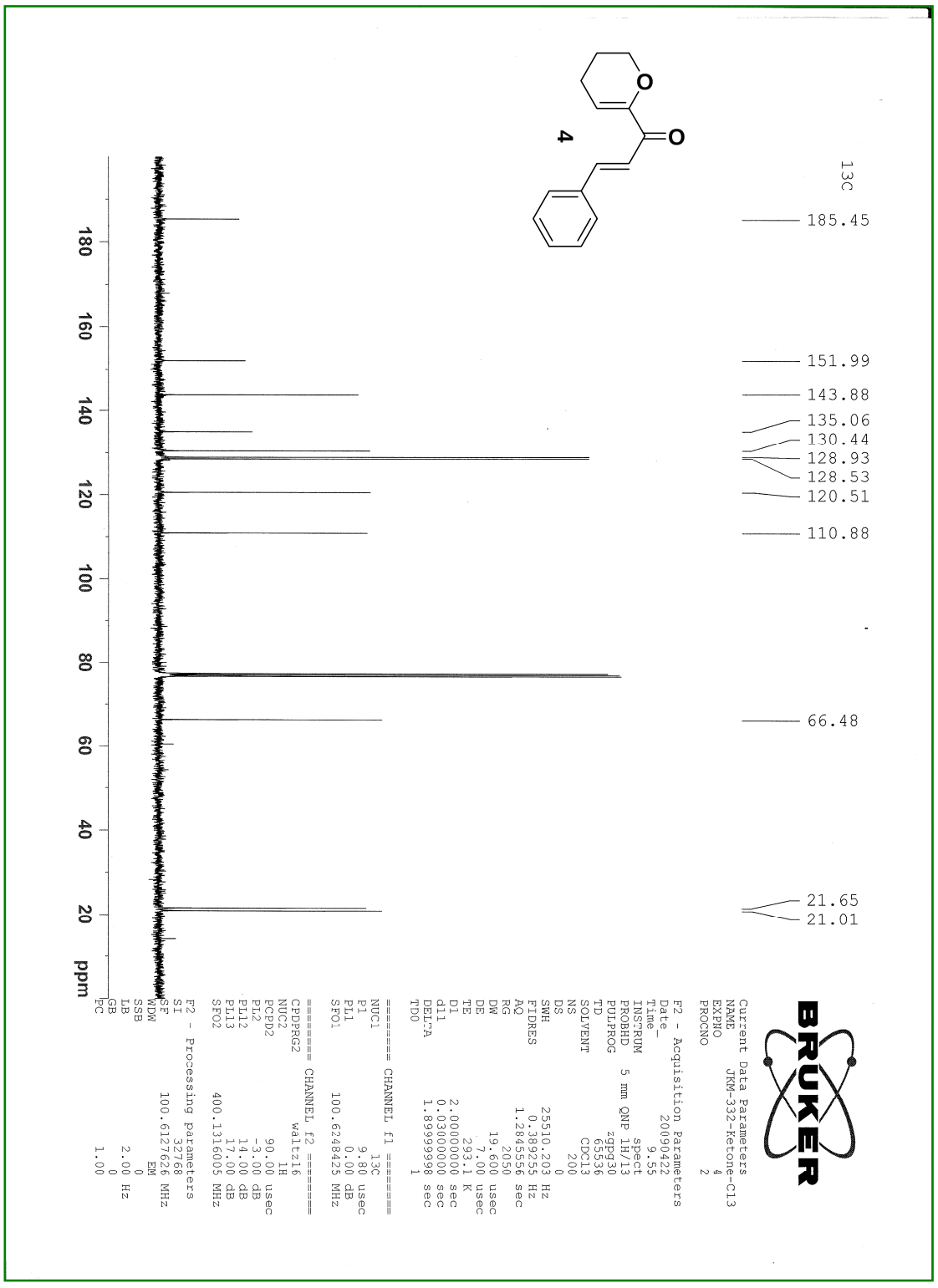
^{1}NMR, ^{13}C NMR and IR Spectra

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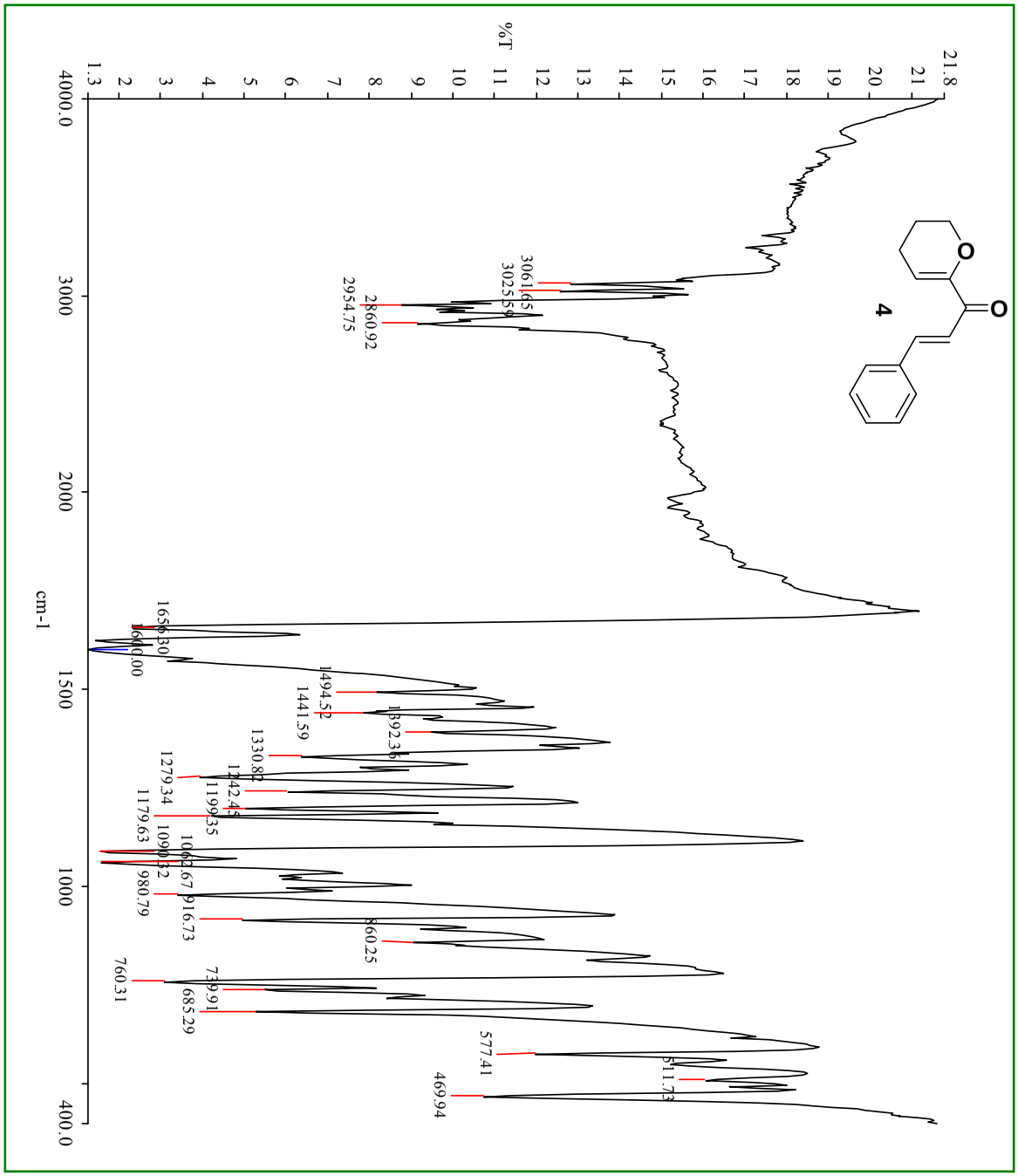
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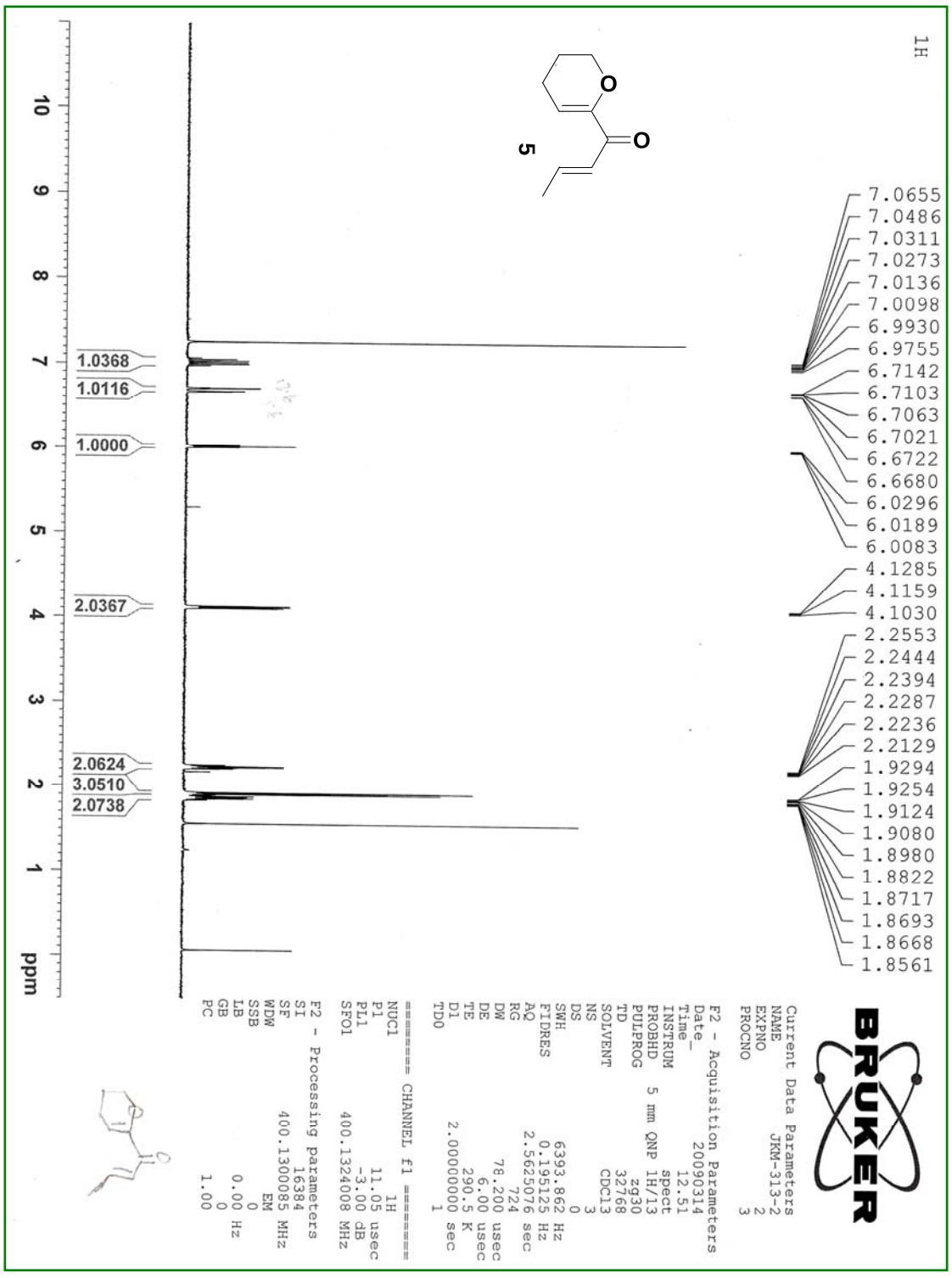
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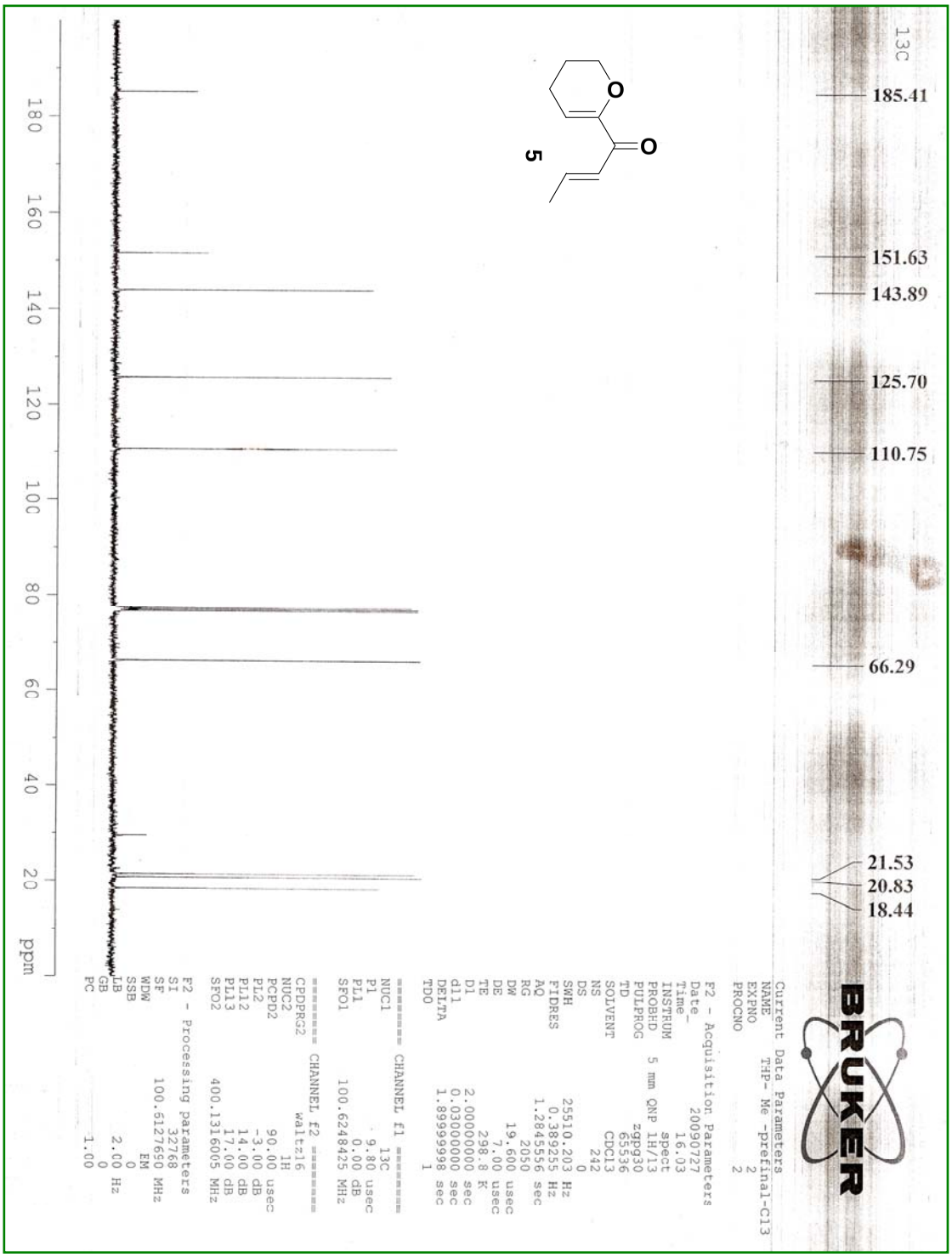
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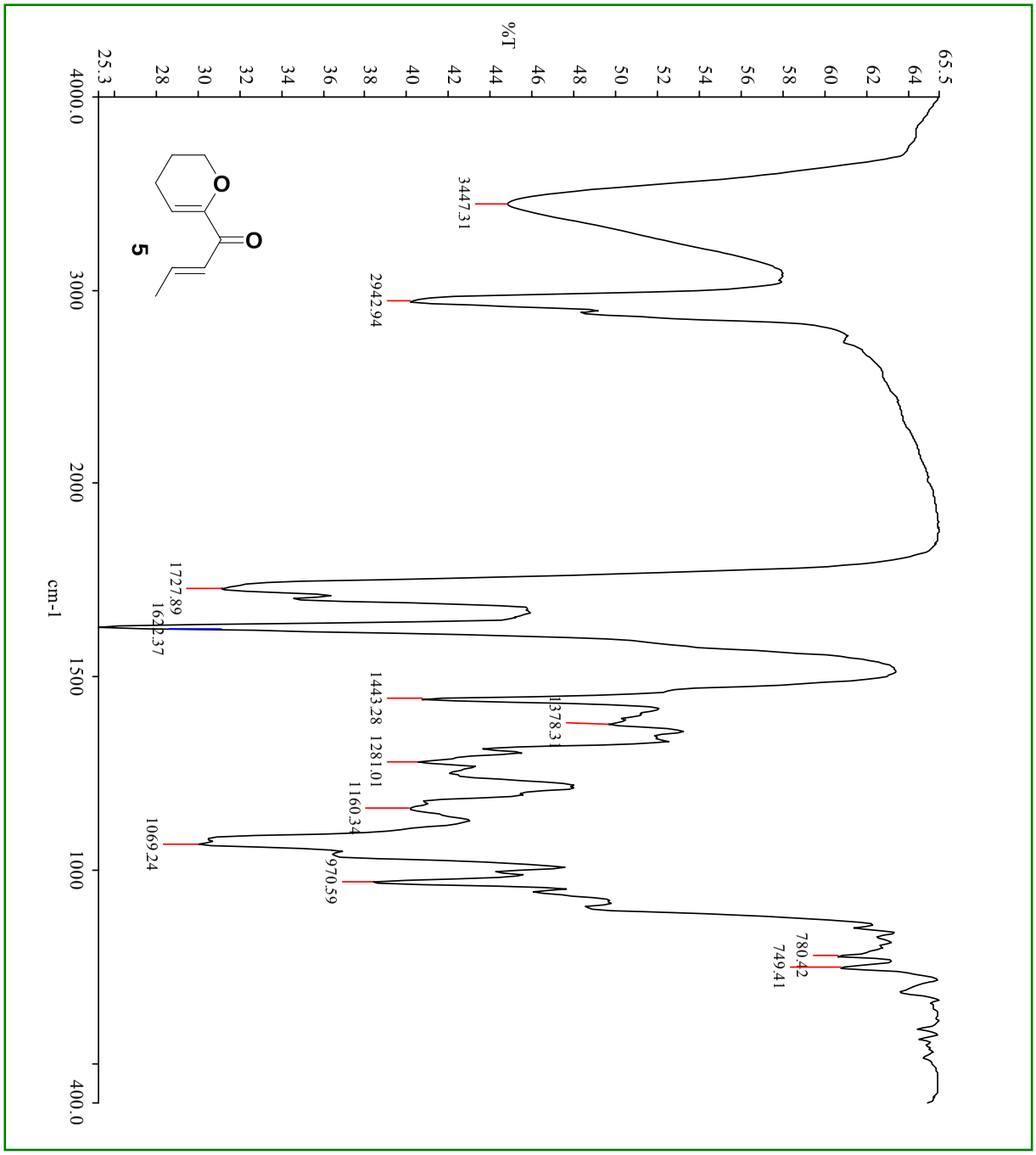
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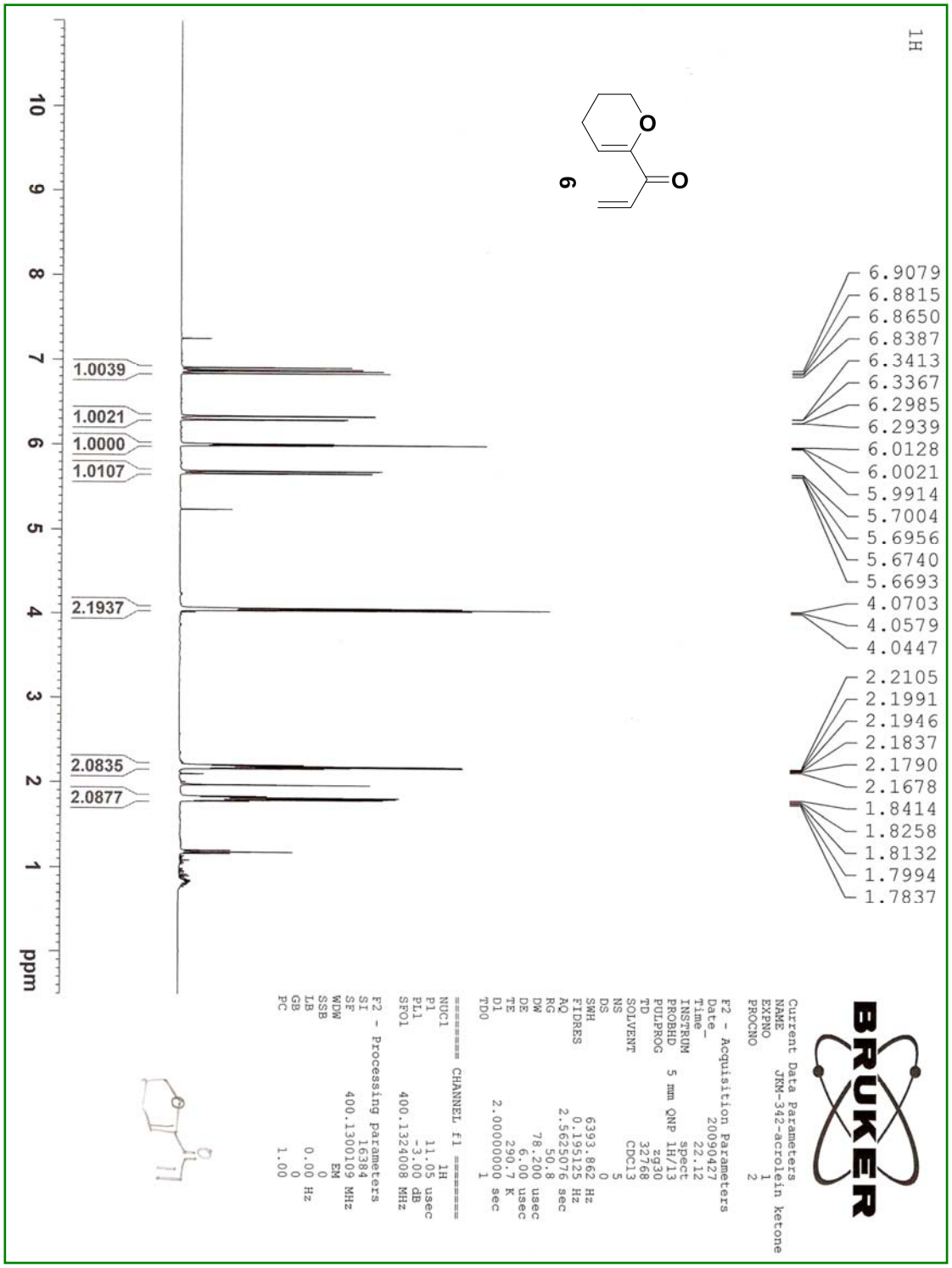
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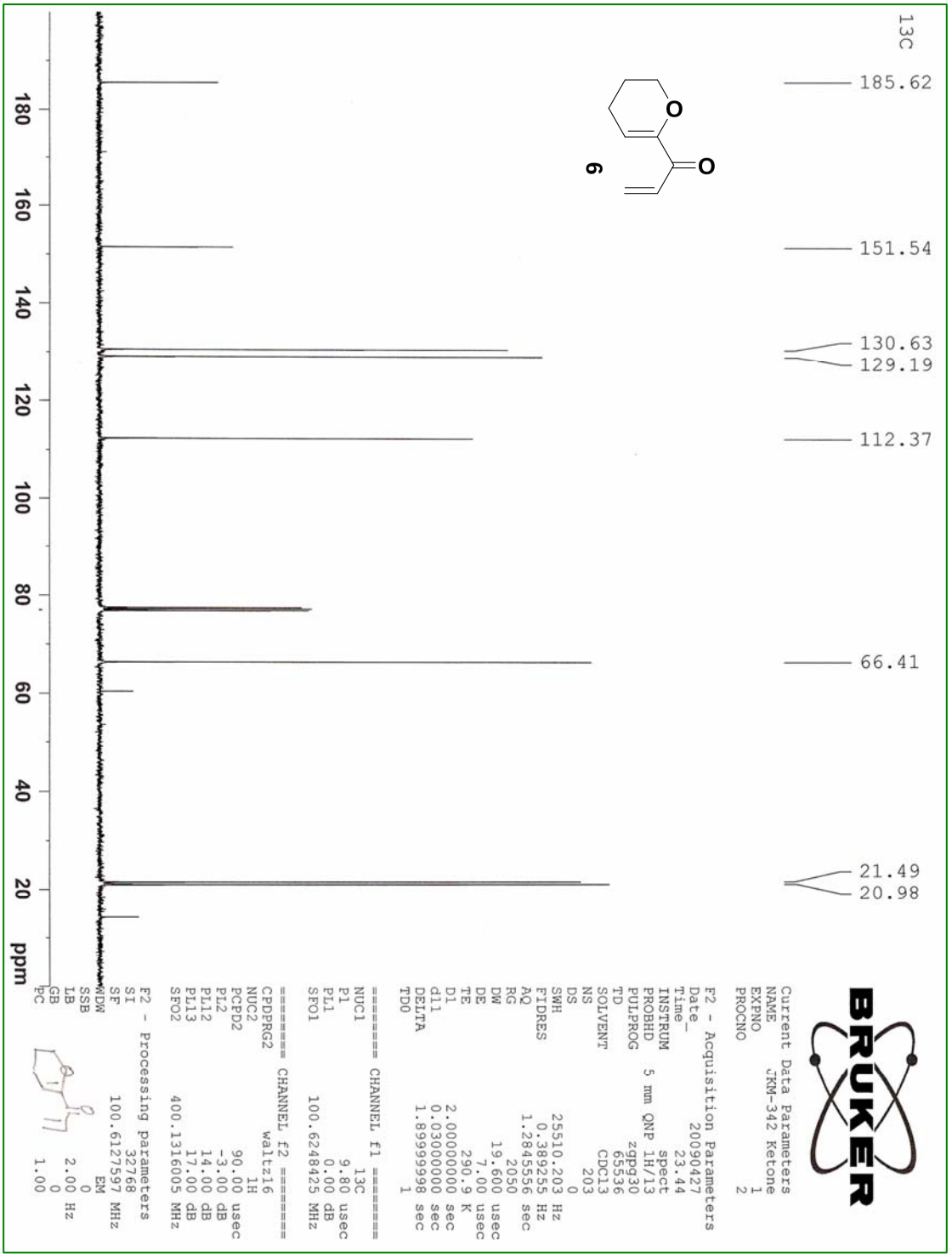
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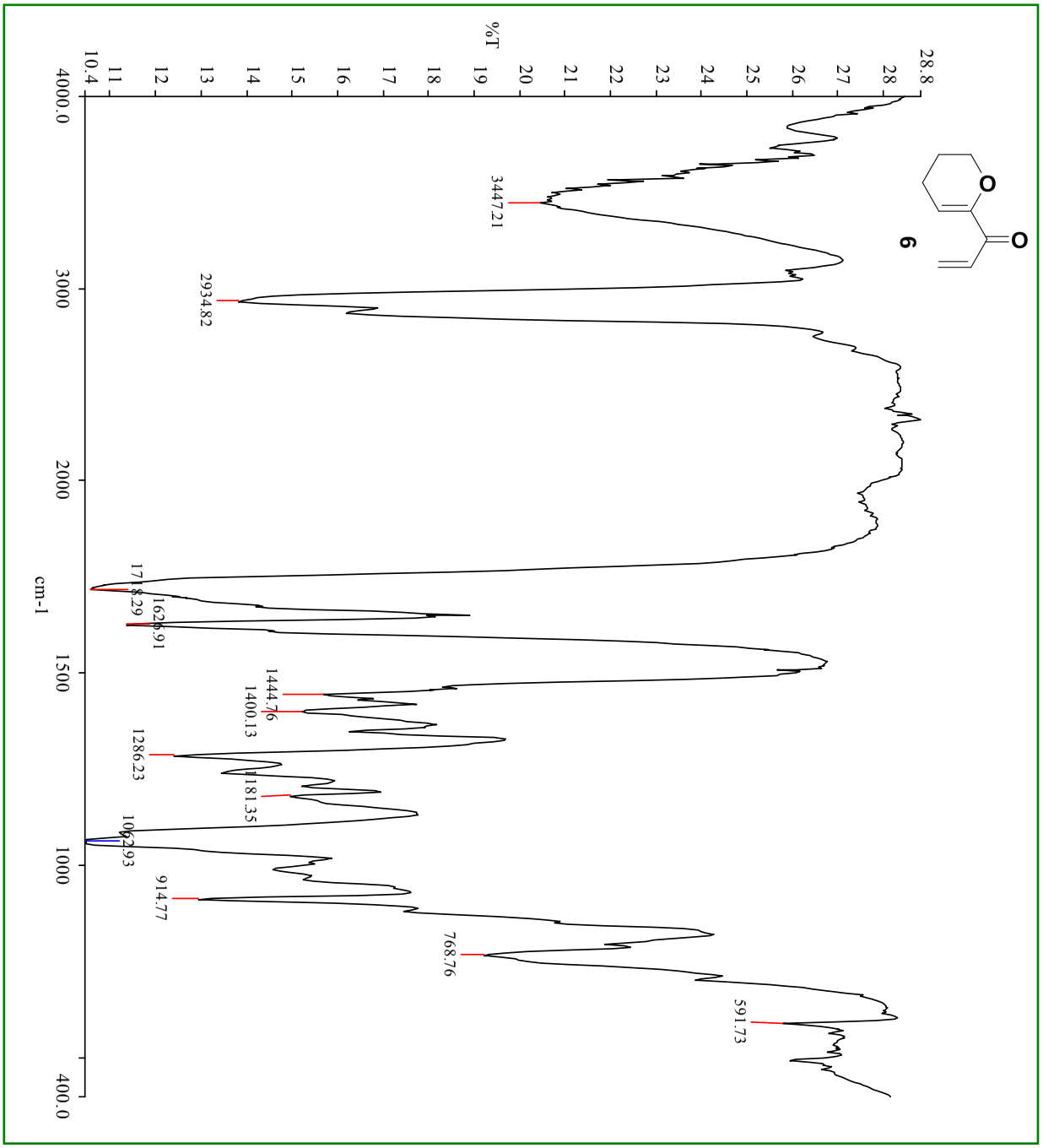
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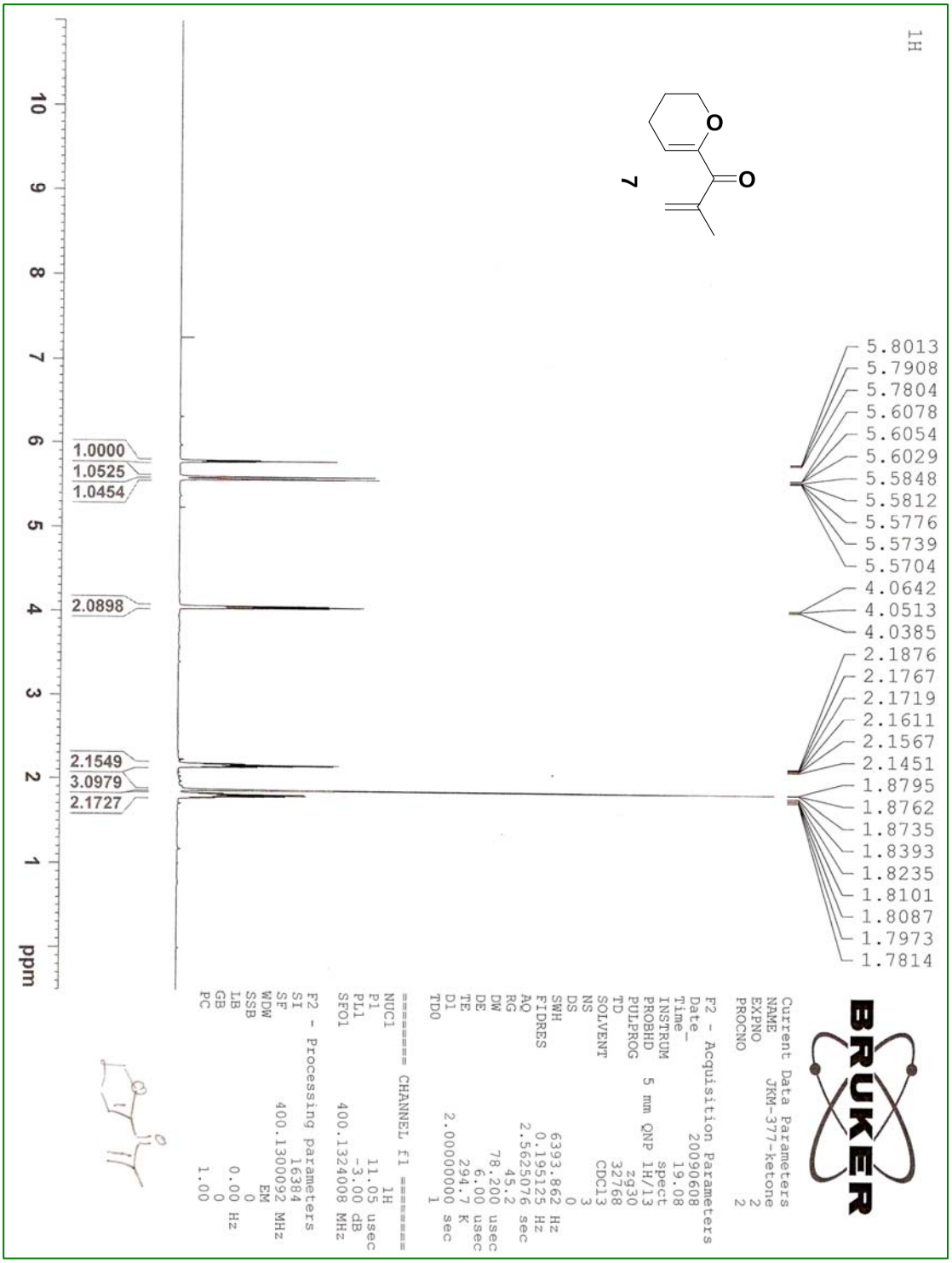
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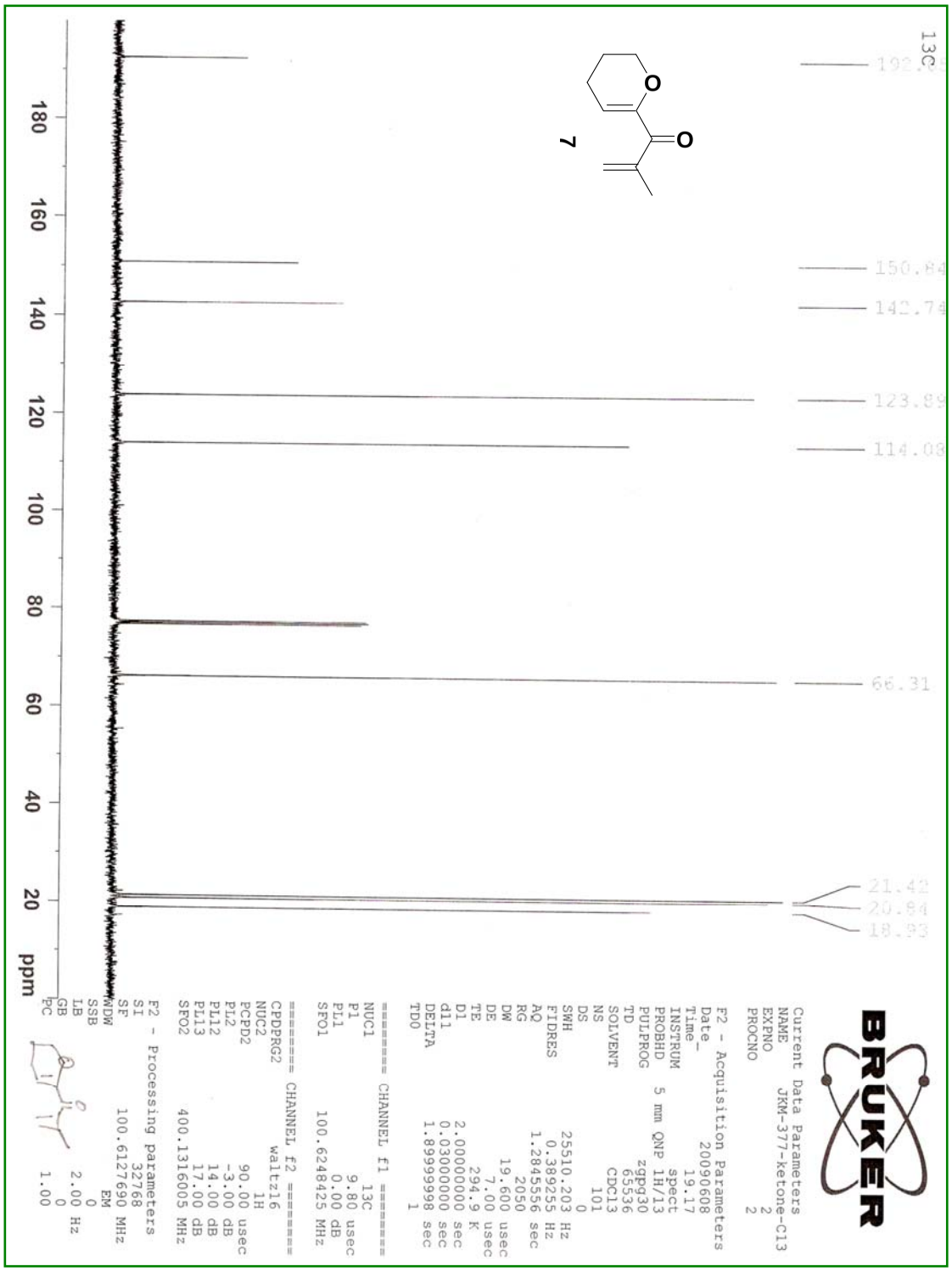
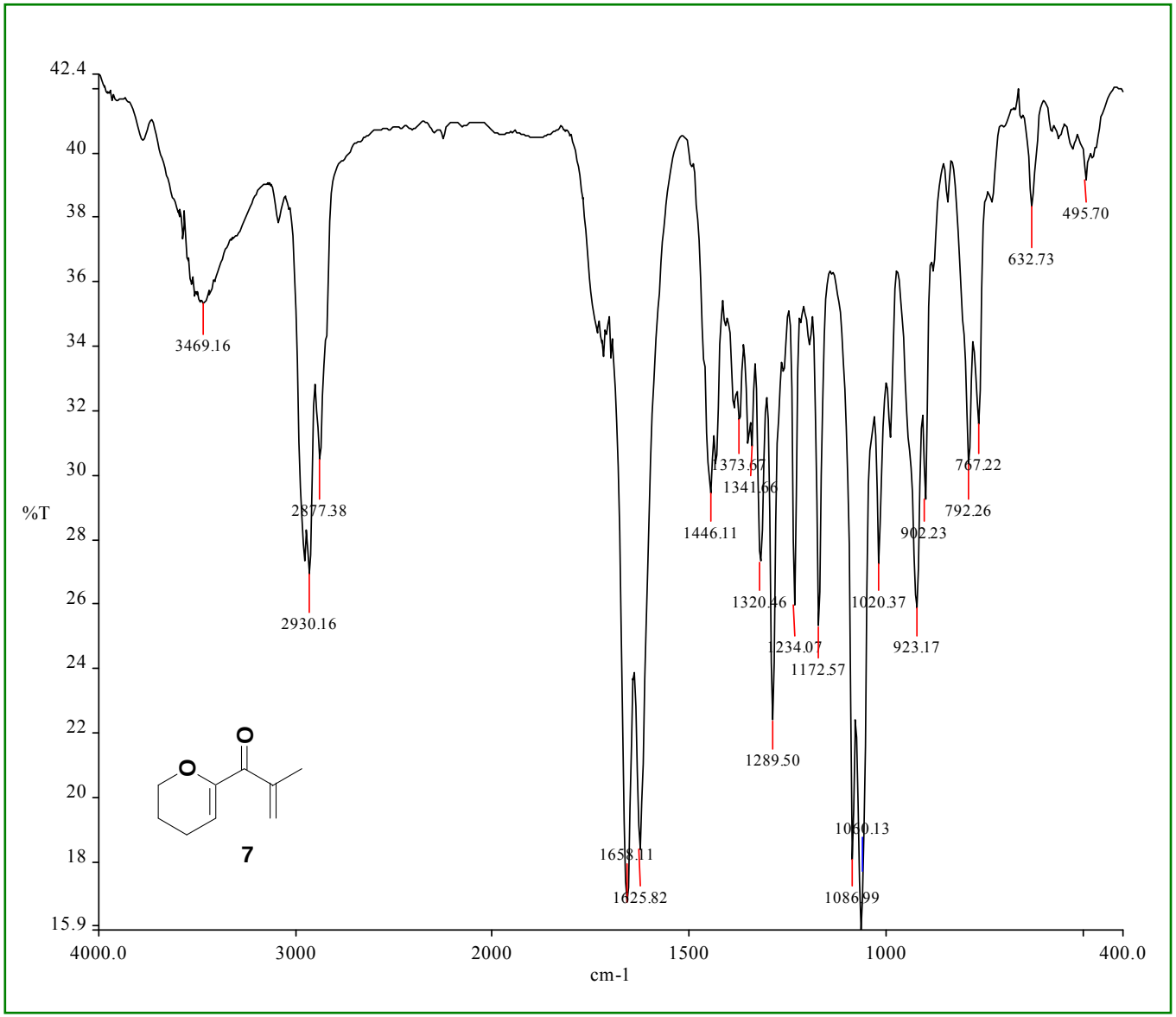
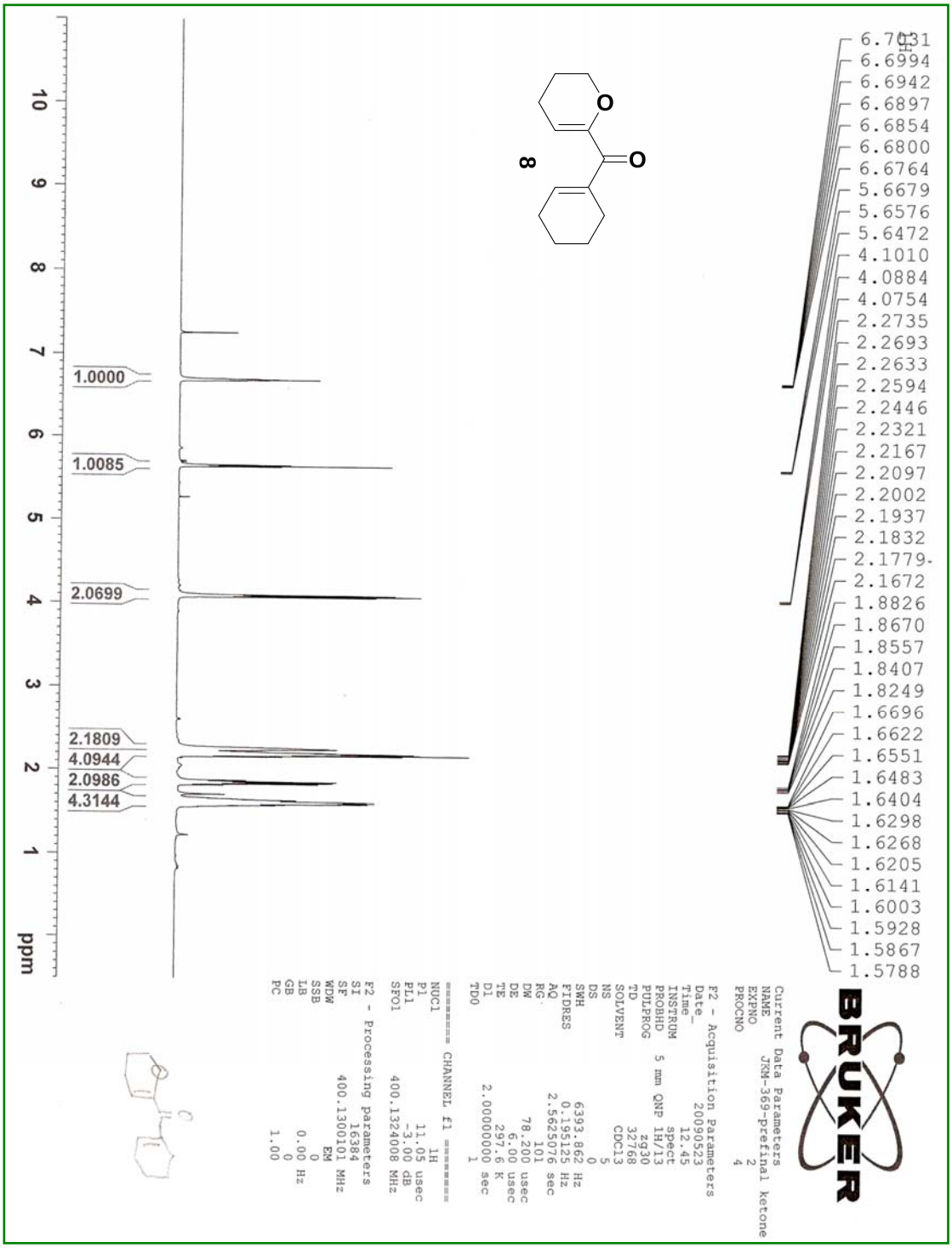
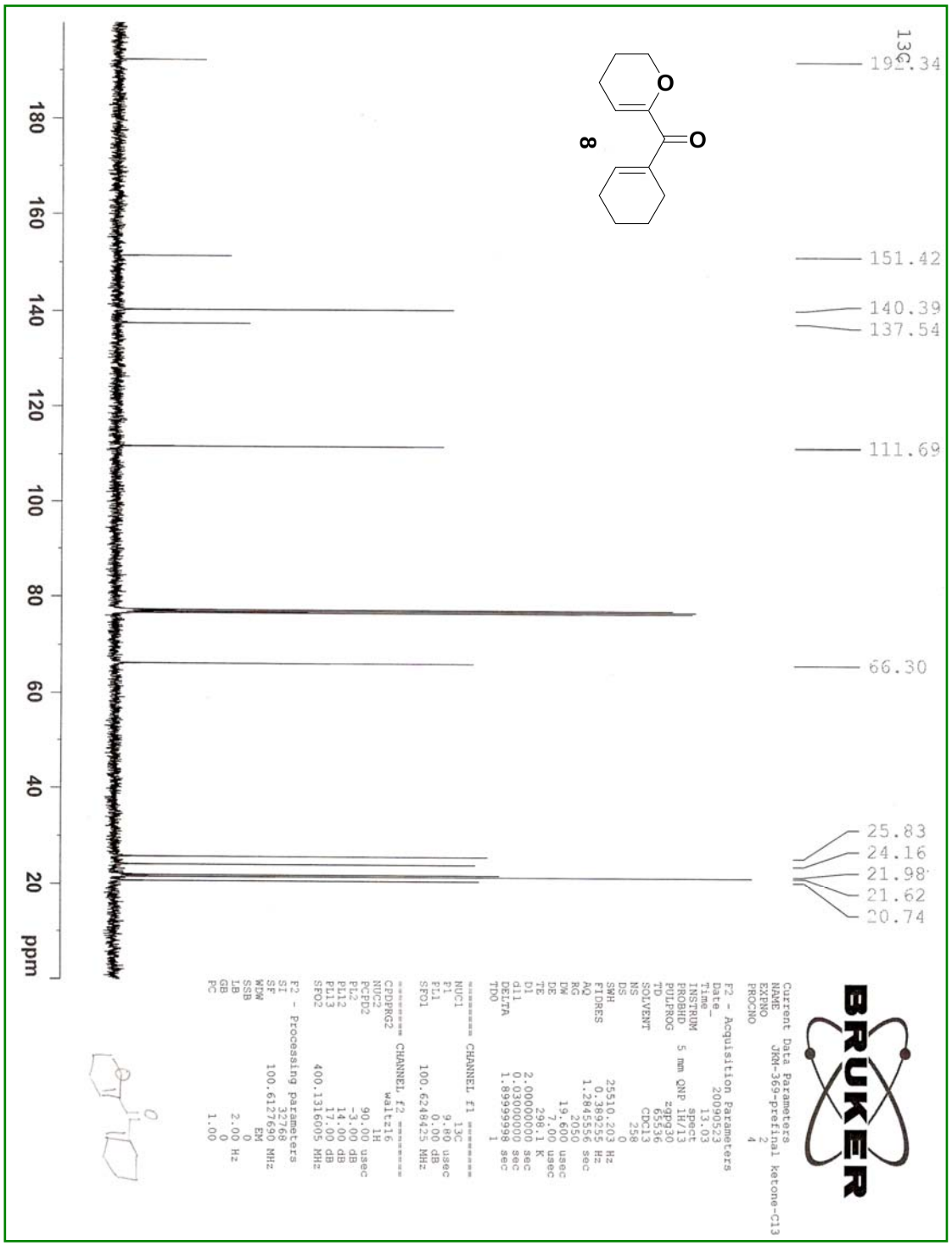
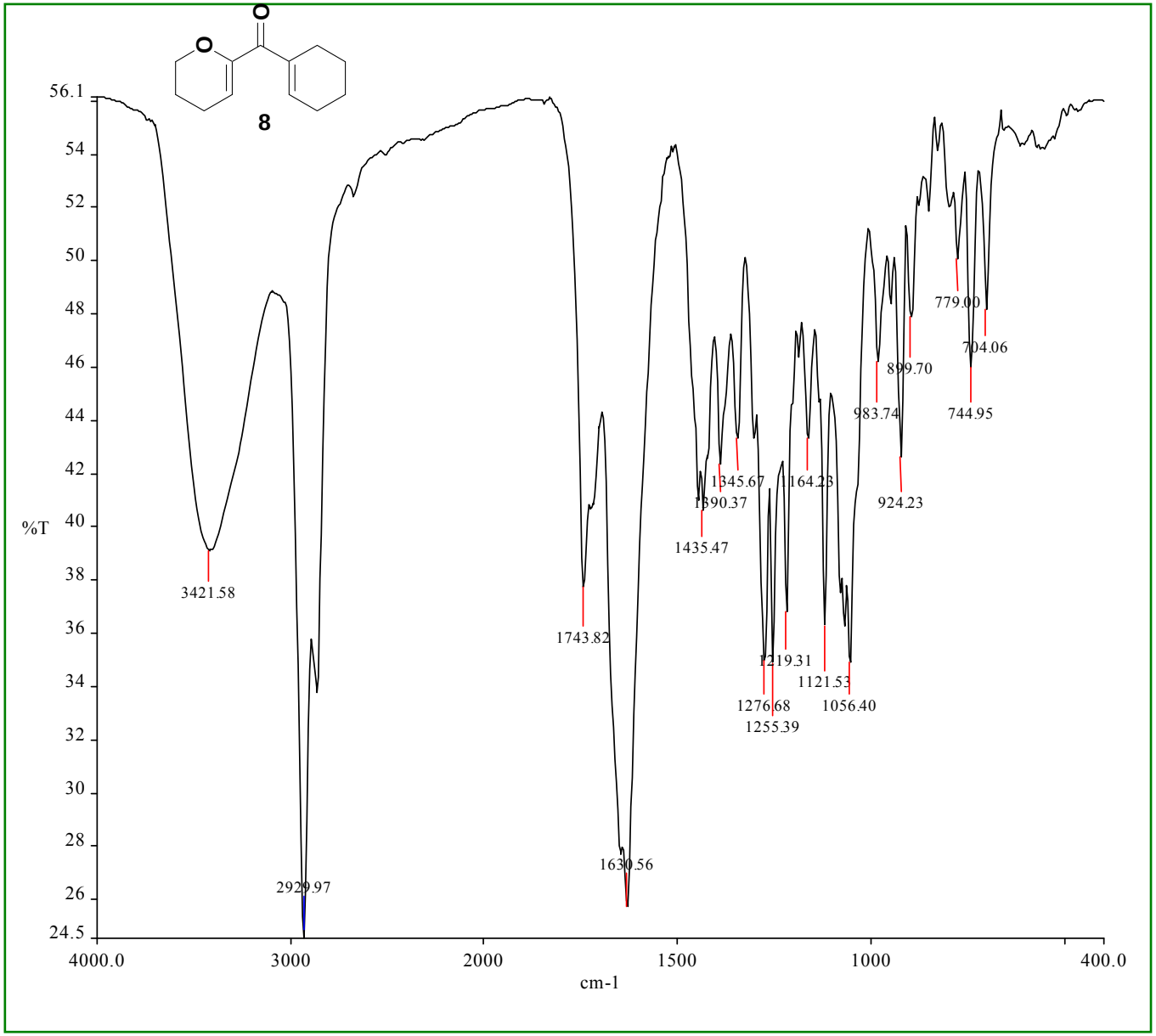
5.5776
5.5739
5.5704
4.0642
4.0513
4.0385
2.1876
2.1767
2.1719
2.1611
2.1567
2.1451
1.8795
1.8762
1.8735
1.8393
1.8235
1.8101
1.8087
1.7973
1.7814
1.0000
1.0525
1.0454
2.0898
2.1549
3.0979
2.1727
ppm
BRUKER
Current Data Parameters
NAME JKM-377-ketone
EXPNO 2
PROCNO 2
F2 - Acquisition Parameters
Date_ 20090608
Time_ 19.08
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 3
DS 0
SWH 6393.862 Hz
FIDRES 0.195125 Hz
AQ 2.5625076 sec
RG 45.2
DW 78.200 usec
DE 6.00 usec
TE 294.7 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.05 usec
PL1 -3.00 dB
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300092 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

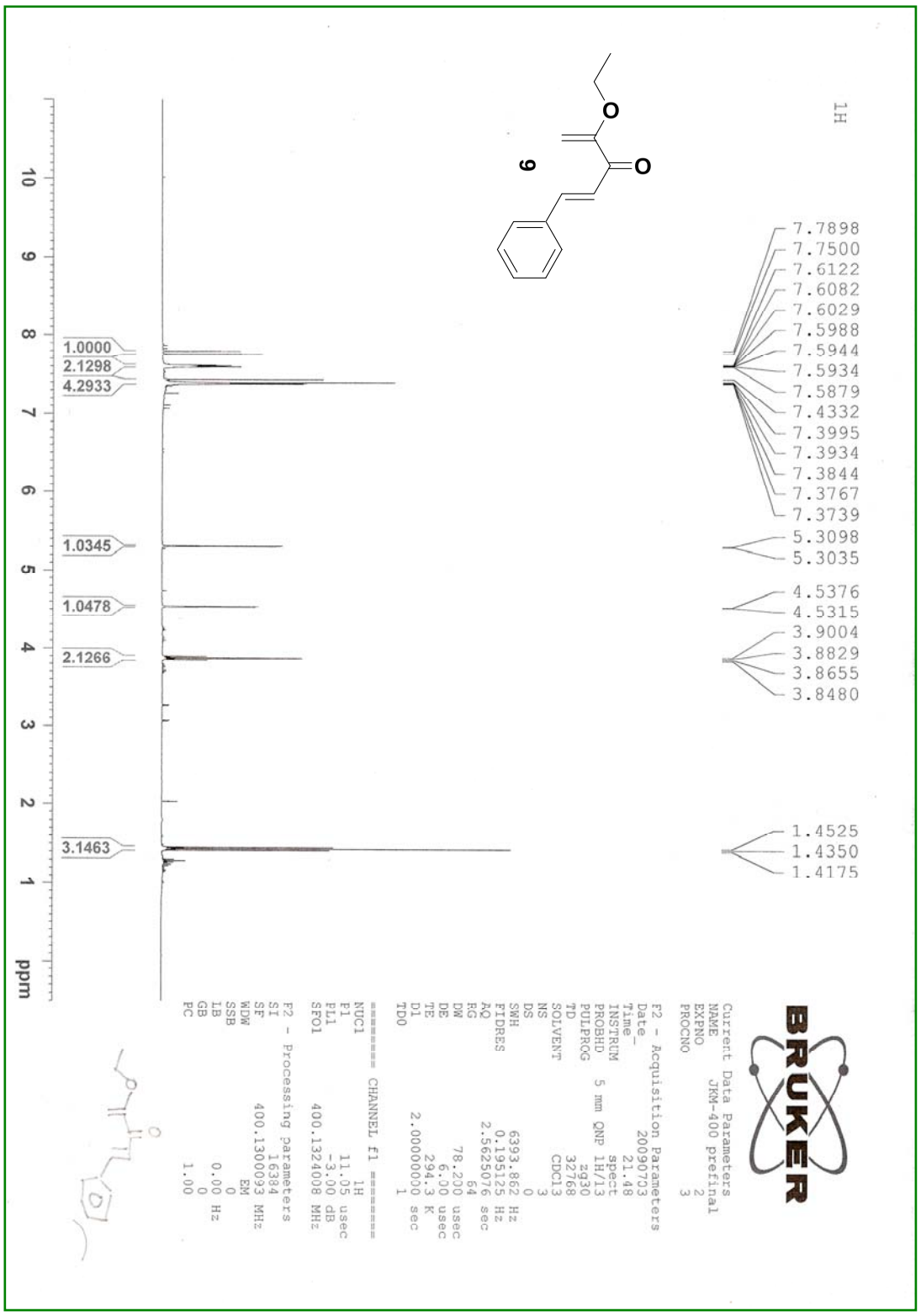

13C
192.5
150.84
142.74
123.89
114.08
66.31
21.42
20.84
18.93
7
O
O
180
160
140
120
100
80
60
40
20
ppm
BRUKER
Current Data Parameters
NAME JKM-377-ketone-C13
EXPNO 2
PROCNO 2
F2 - Acquisition Parameters
Date_ 20090608
Time_ 19.17
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 101
DS 0
SWH 25510.203 Hz
FIDRES 0.389255 Hz
AQ 1.2845556 sec
RG 2050
DW 19.600 usec
DE 7.00 usec
TE 294.9 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.80 usec
PL1 0.00 dB
SFO1 100.6248425 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -3.00 dB
PL12 14.00 dB
PL13 17.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00

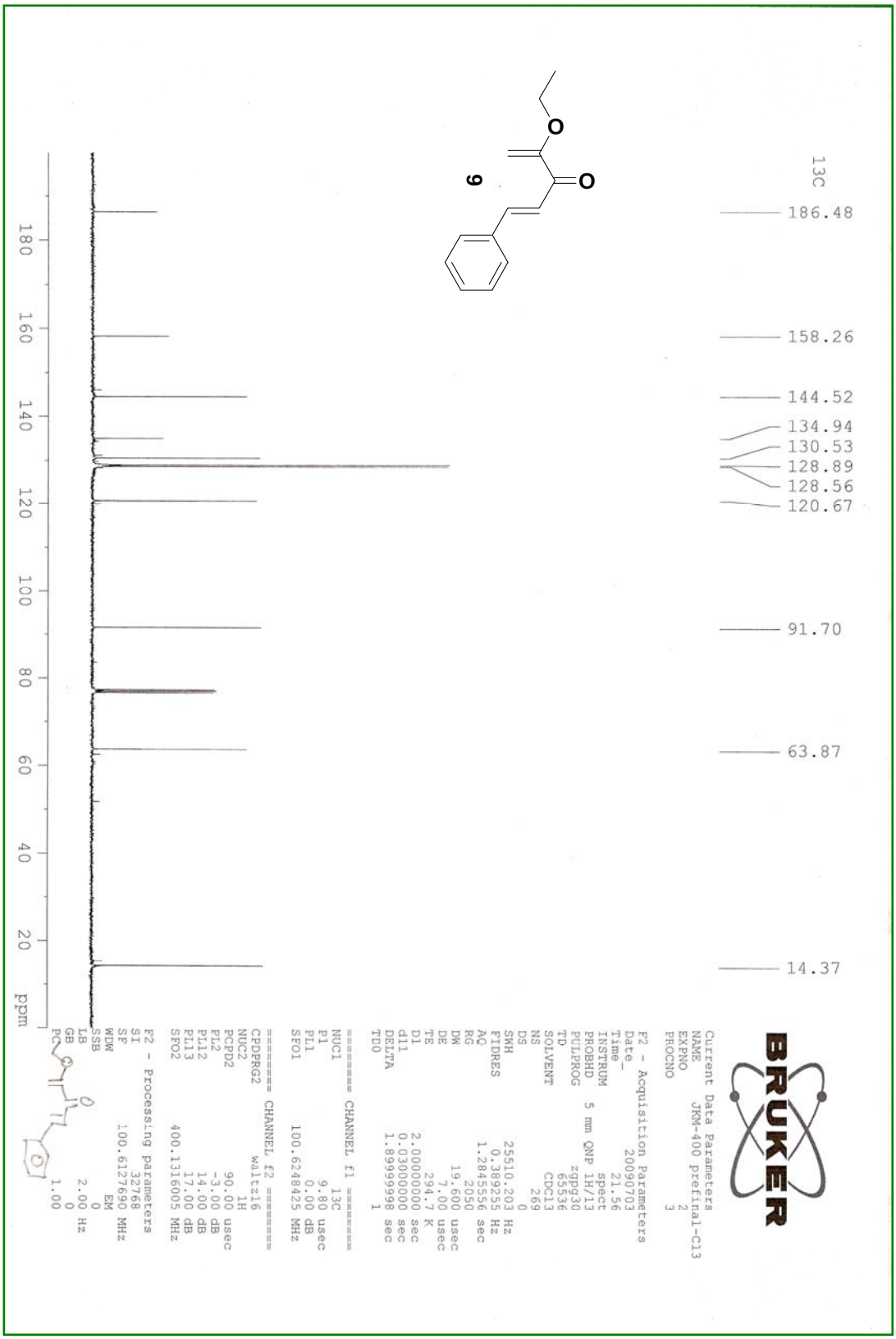


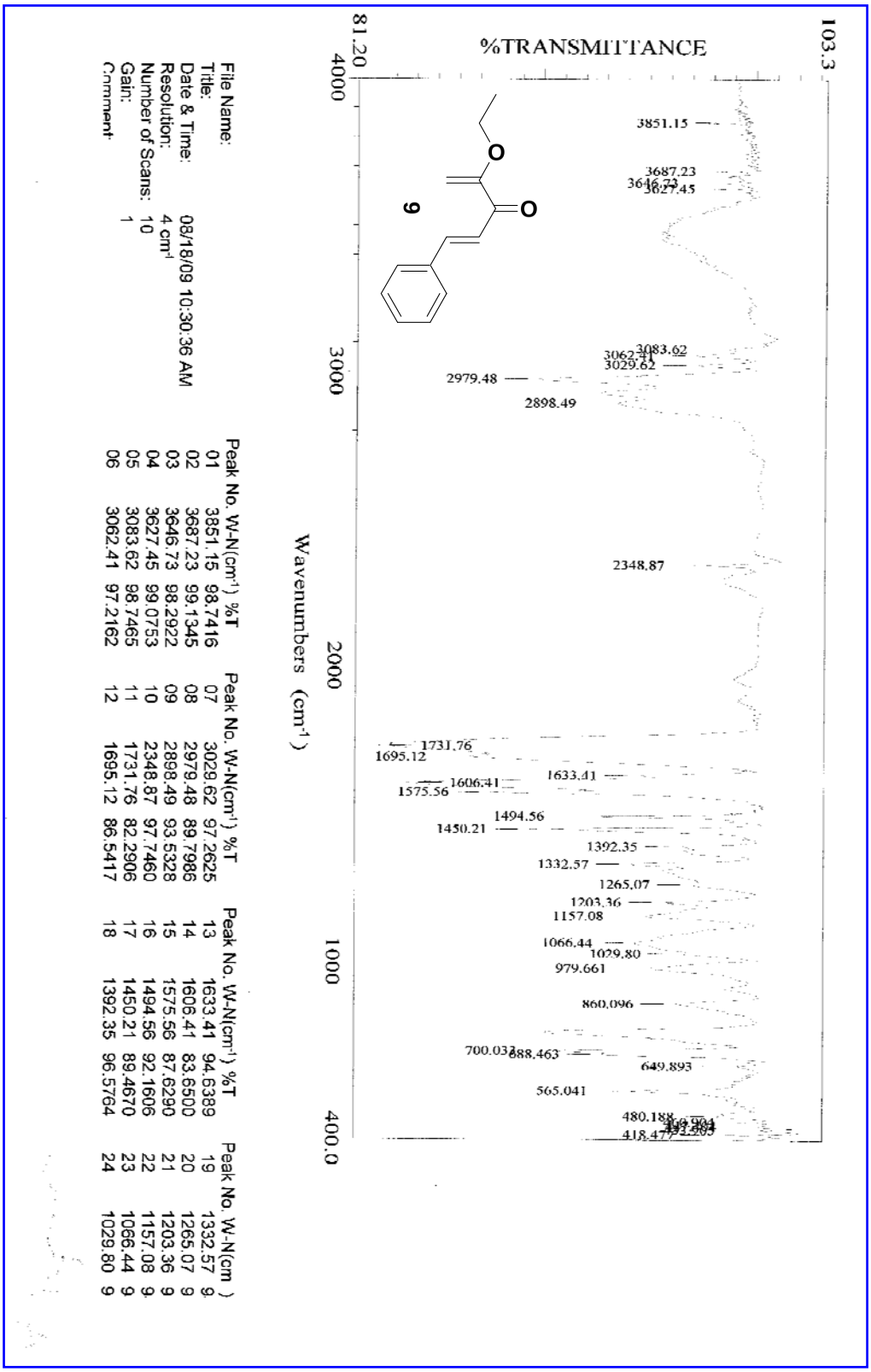

6.7231
6.6994
6.6942
6.6897
6.6854
6.6800
6.6764
5.6679
5.6576
5.6472
4.1010
4.0884
4.0754
2.2735
2.2693
2.2633
2.2594
2.2446
2.2321
2.2167
2.2097
2.2002
2.1937
2.1832
2.1779
2.1672
1.8826
1.8670
1.8557
1.8407
1.8249
1.6696
1.6622
1.6551
1.6483
1.6404
1.6298
1.6268
1.6205
1.6141
1.6003
1.5928
1.5867
1.5788
O
O
8
1.0000
1.0085
2.0699
2.1809
4.0944
2.0986
4.3144
ppm
BRUKER
Current Data Parameters
NAME JKM-369-prefinal ketone
EXPNO 2
PROCNO 4
F2 - Acquisition Parameters
Date_ 20090523
Time_ 12.45
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 5
DS 0
SWH 6393.862 Hz
FIDRES 0.195125 Hz
AQ 2.5625076 sec
RG 101
DW 78.200 usec
DE 6.00 usec
TE 297.6 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.05 usec
PL1 -3.00 dB
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300101 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00


13C
191.34
151.42
140.39
137.54
111.69
66.30
25.83
24.16
21.98
21.62
20.74
O
O
8
180
160
140
120
100
80
60
40
20
ppm
BRUKER
Current Data Parameters
NAME JRM-369-prefinal ketone-C13
EXPNO 2
PROCNO 4
F2 - Acquisition Parameters
Date_ 20090303
Time 13.03
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 0
SWH 25510.203 Hz
FIDRES 0.389255 Hz
AQ 1.2845556 sec
RG 1.3050
DW 19.600 usec
DE 7.00 usec
TE 298.1 K
D1 2.00000000 sec
D11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.80 usec
PL1 0.00 dB
SFO1 100.6248425 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -3.00 dB
PL12 14.00 dB
PL13 17.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00



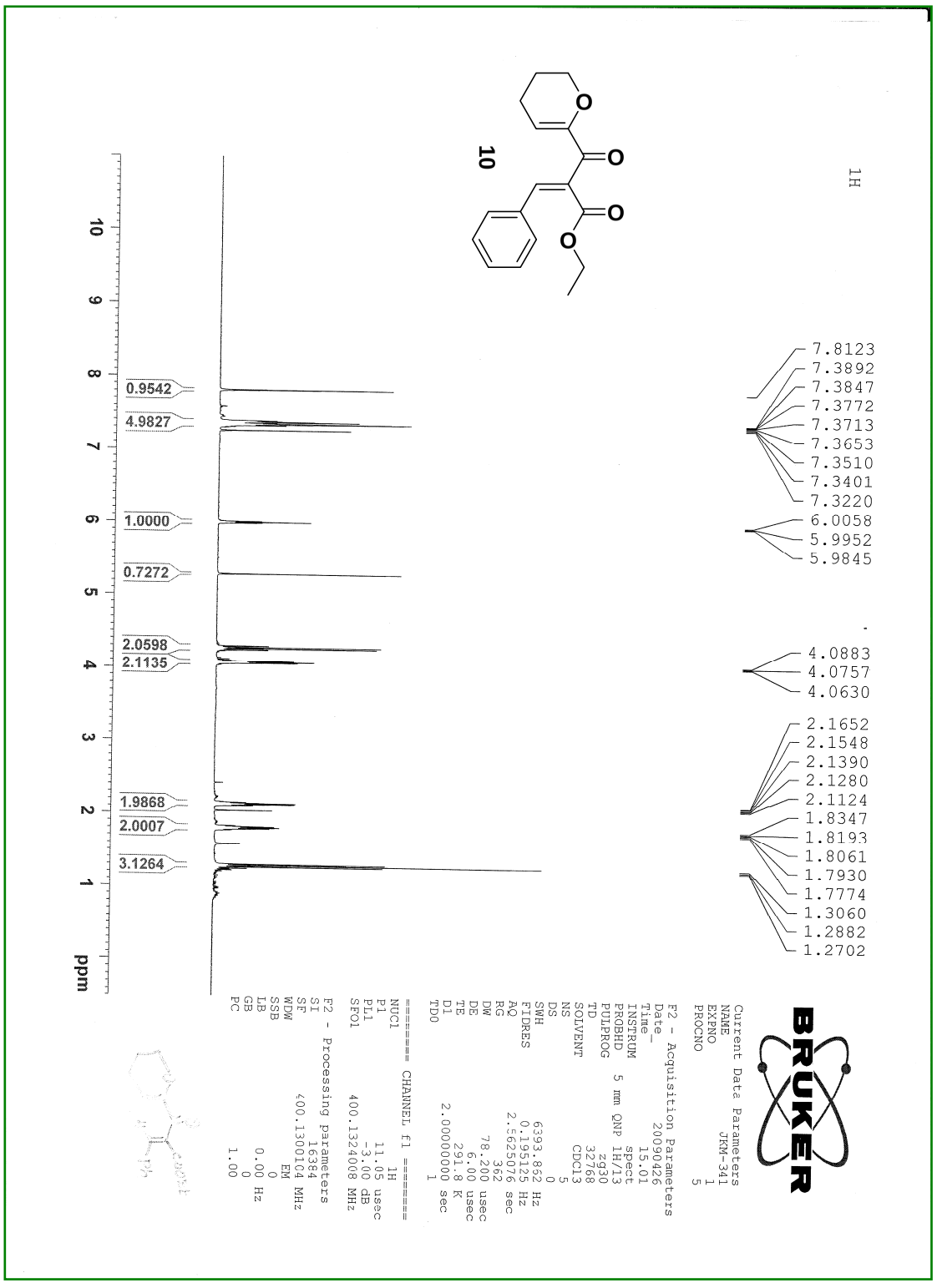

1H
9
O
O
7.7898
7.7500
7.6122
7.6082
7.6029
7.5988
7.5944
7.5934
7.5879
7.4332
7.3995
7.3934
7.3844
7.3767
7.3739
5.3098
5.3035
4.5376
4.5315
3.9004
3.8829
3.8655
3.8480
1.4525
1.4350
1.4175
1.0000
2.1298
4.2933
1.0345
1.0478
2.1266
3.1463
ppm
BRUKER
Current Data Parameters
NAME JKM-400 prefinal
EXPNO 2
PROCNO 3
F2 - Acquisition Parameters
Date_ 20090703
Time_ 21.48
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 3
DS 0
SWH 6393.862 Hz
FIDRES 0.195125 Hz
AQ 2.5625076 sec
RG 64
DW 78.200 usec
DE 6.00 usec
TE 294.3 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.05 usec
PL1 -3.00 dB
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300093 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

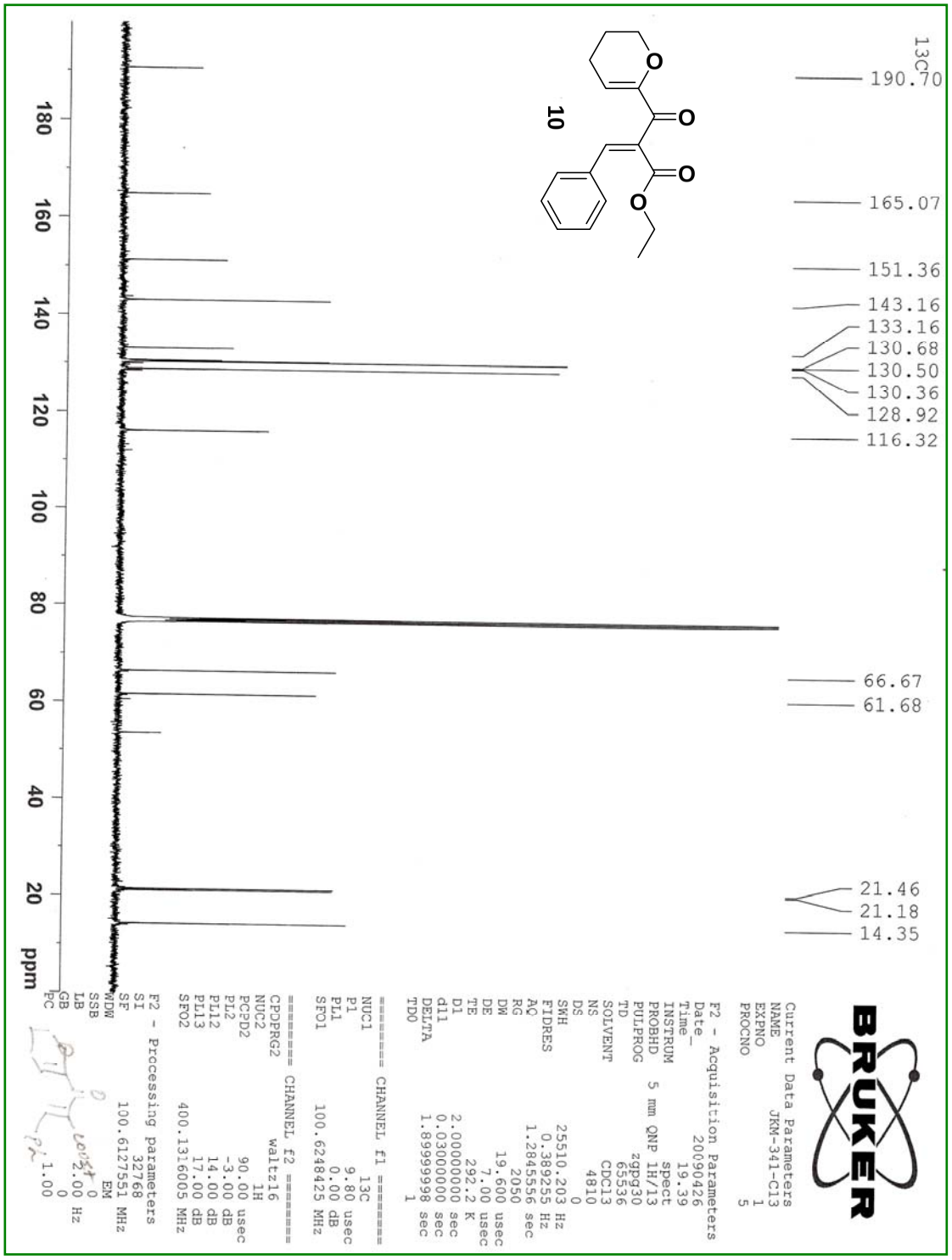

13C
186.48
158.26
144.52
134.94
130.53
128.89
128.56
120.67
91.70
63.87
14.37
O
O
6
ppm
180
160
140
120
100
80
60
40
20
BRUKER
Current Data Parameters
NAME JKM-400 prefinal-C13
EXPNO 2
PROCNO 3
F2 - Acquisition Parameters
Date_ 20090703
Time_ 21.56
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 269
DS 0
SWH 25510.203 Hz
FIDRES 0.389255 Hz
AQ 1.2845556 sec
RG 2050
DW 19.600 usec
DE 7.00 usec
TE 294.7 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.80 usec
PL1 0.00 dB
SFO1 100.6248425 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -3.00 dB
PL12 14.00 dB
PL13 17.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00

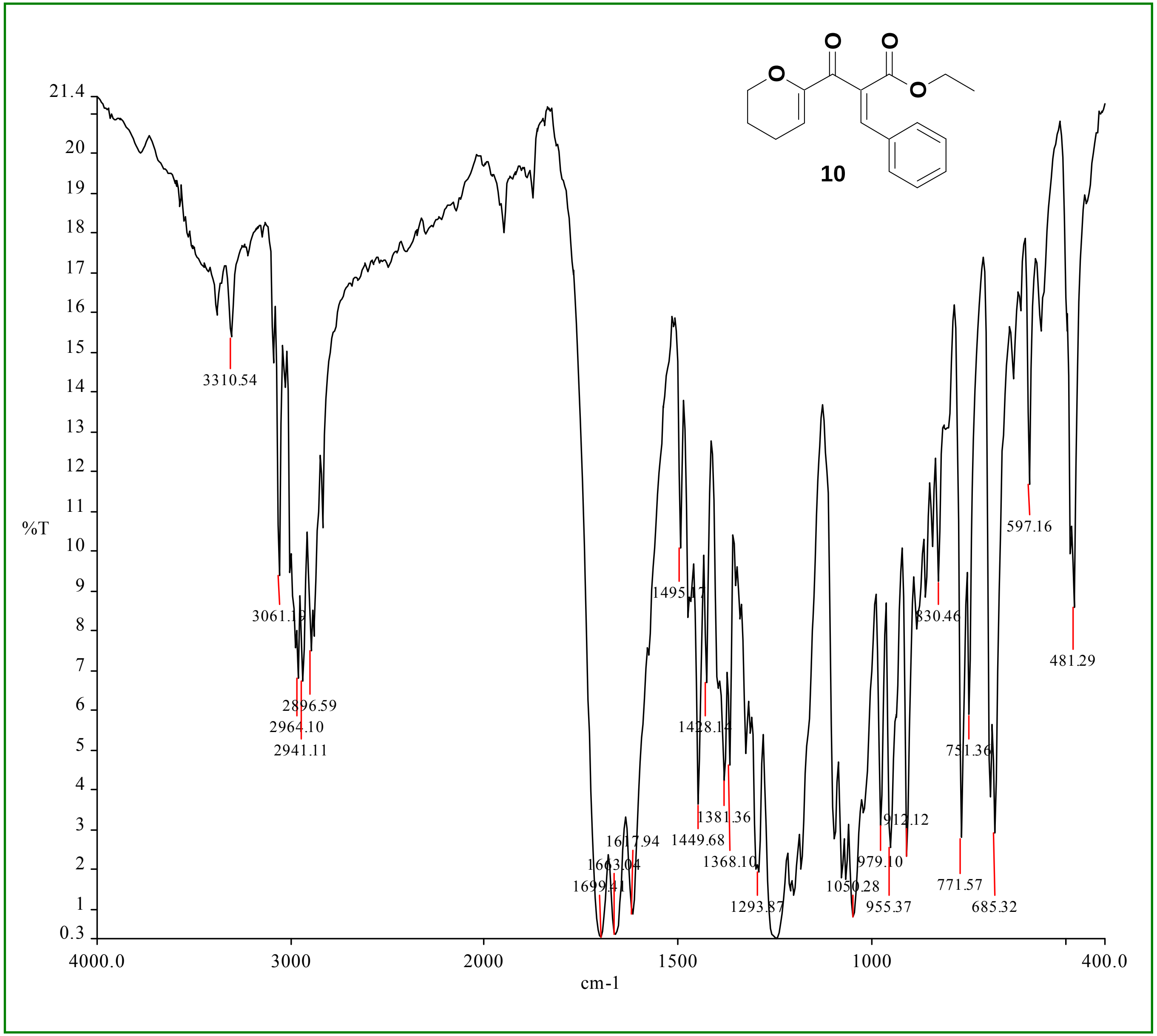


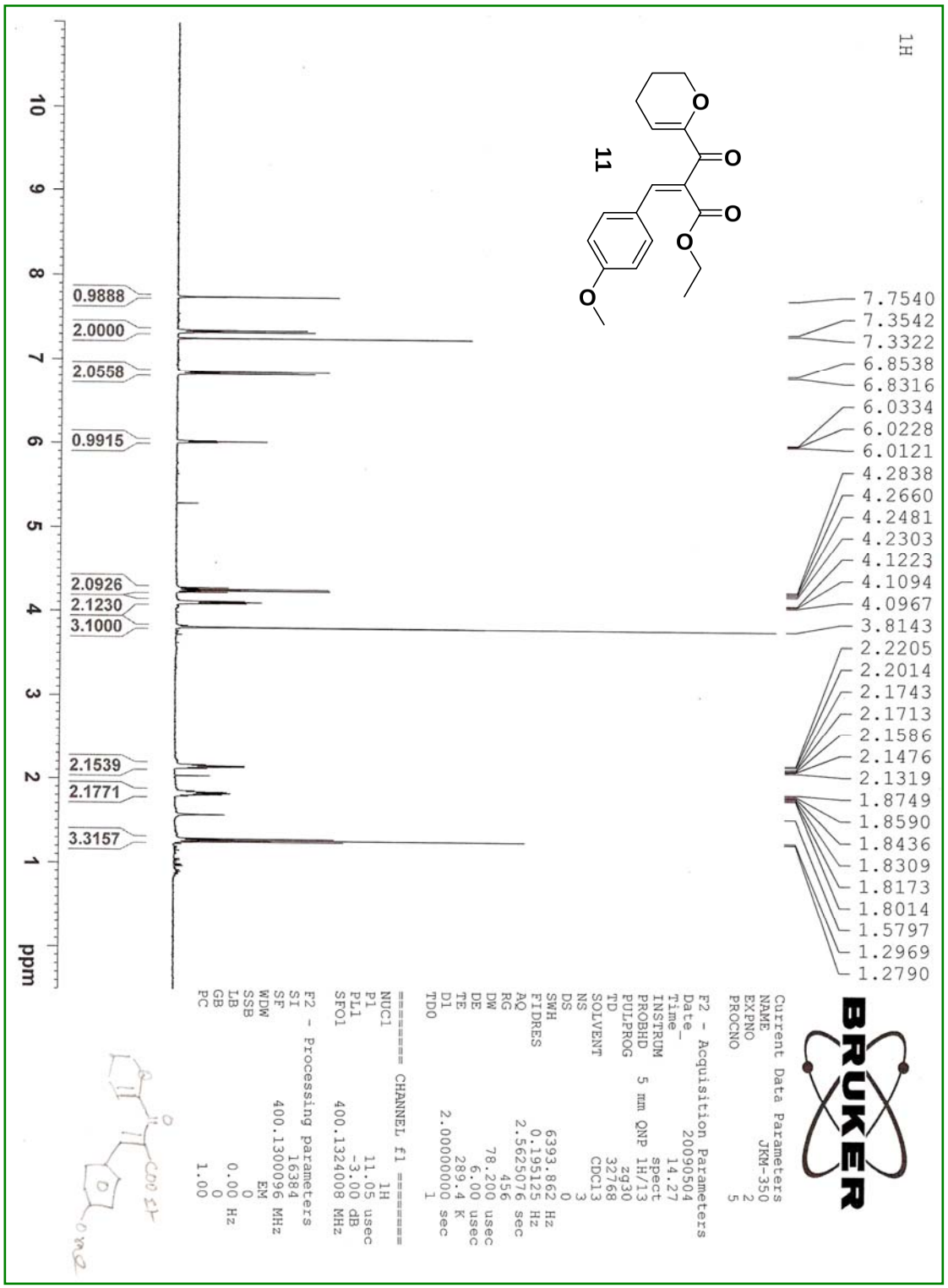
File Name:
Title:
Date & Time: 08/18/09 10:30:36 AM
Resolution: 4 cm-1
Number of Scans: 10
Gain: 1
Comment:

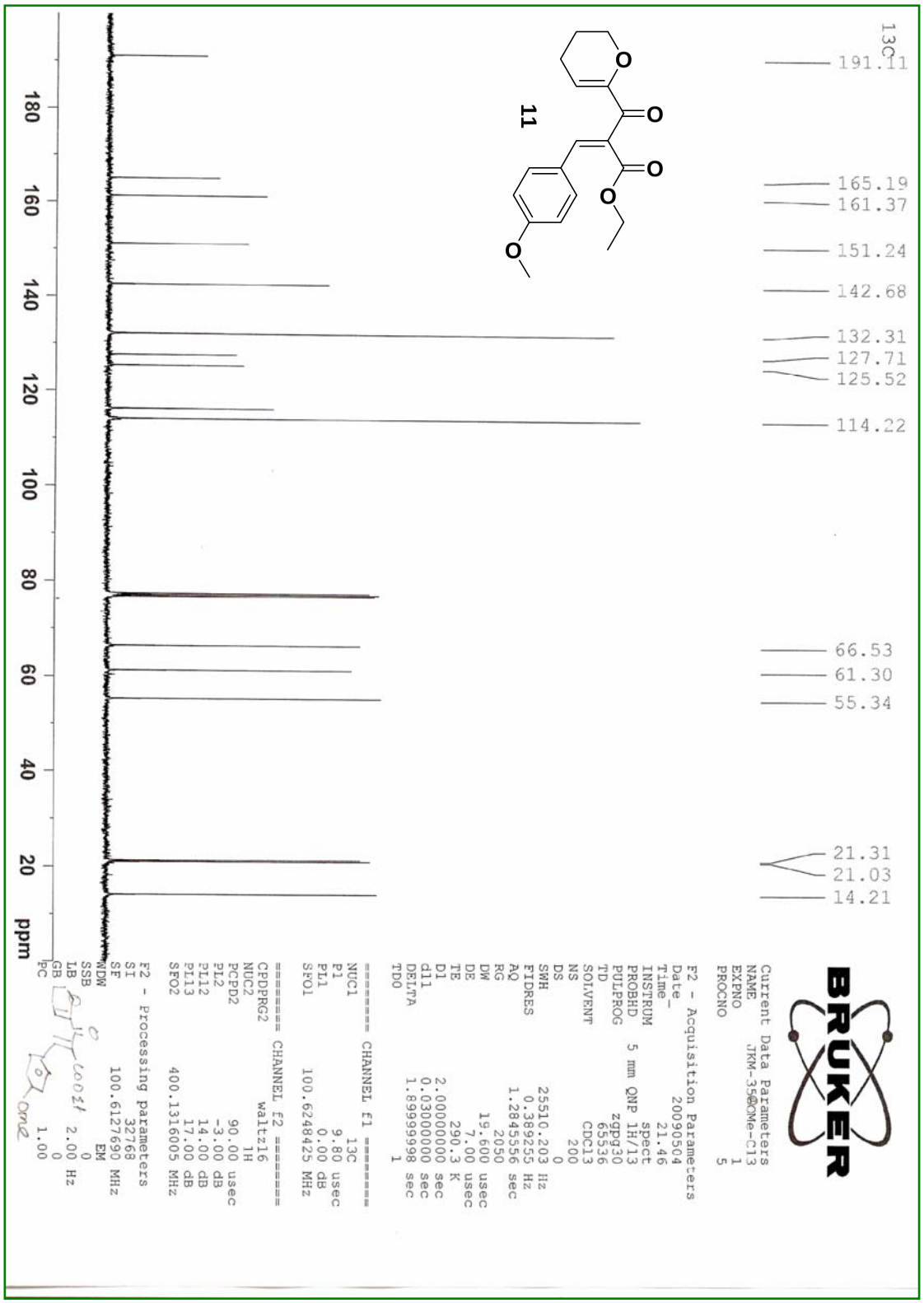
Peak No.	W-N(cm-1)	%T
01	3851.15	98.7416
02	3687.23	99.1345
03	3646.73	98.2922
04	3627.45	99.0753
05	3083.62	98.7465
06	3062.41	97.2162
07	3029.62	97.2625
08	2979.48	89.7986
09	2898.49	93.5328
10	2348.87	97.7460
11	1731.76	82.2906
12	1695.12	86.5417
13	1633.41	94.6389
14	1606.41	83.6500
15	1575.56	87.6290
16	1494.56	92.1606
17	1450.21	89.4670
18	1392.35	96.5764
19	1332.57	9
20	1265.07	9
21	1203.36	9
22	1157.08	9
23	1066.44	9
24	1029.80	9

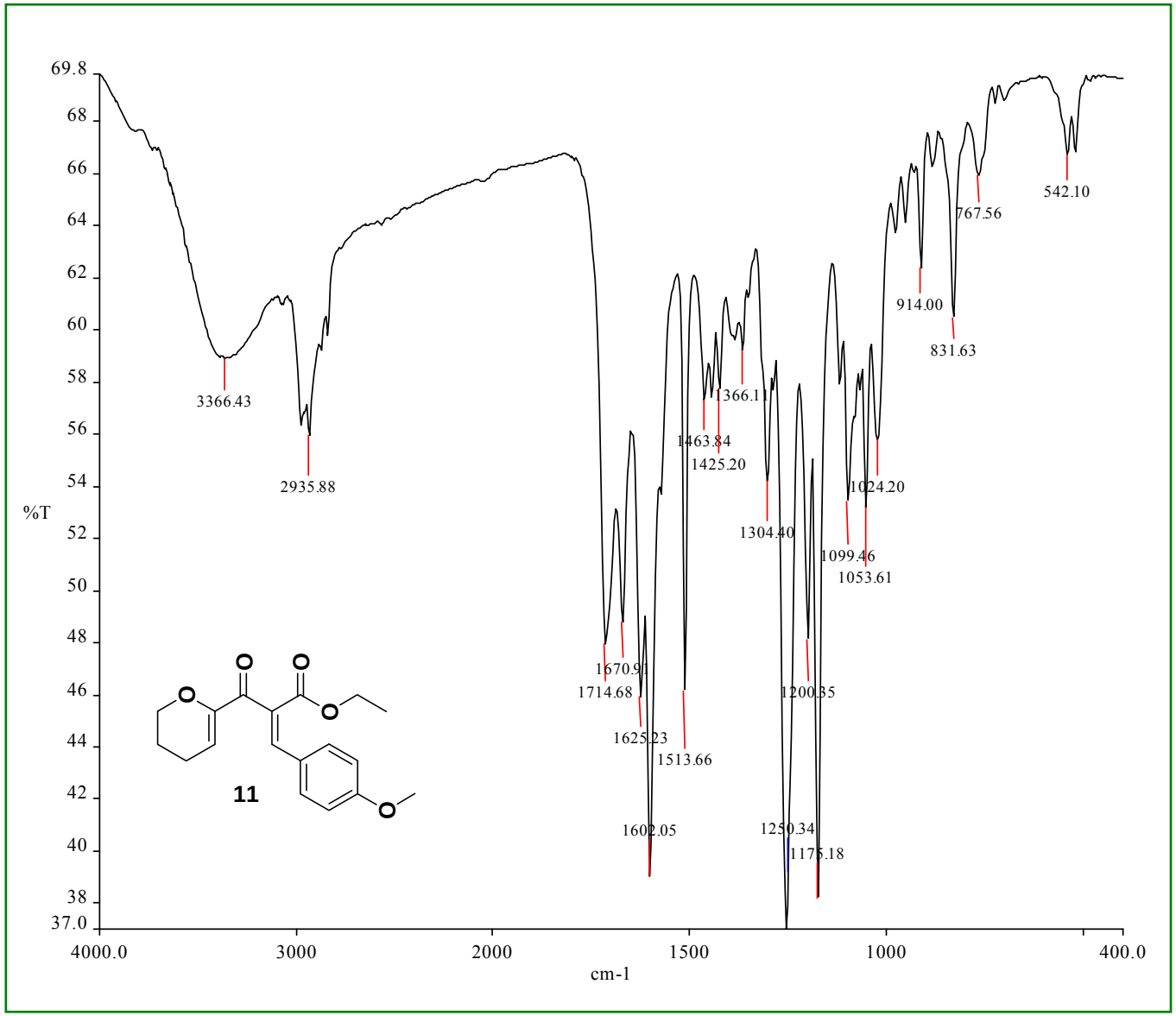

1H
10
7.8123
7.3892
7.3847
7.3772
7.3713
7.3653
7.3510
7.3401
7.3220
6.0058
5.9952
5.9845
4.0883
4.0757
4.0630
2.1652
2.1548
2.1390
2.1280
2.1124
1.8347
1.8193
1.8061
1.7930
1.7774
1.3060
1.2882
1.2702
0.9542
4.9827
1.0000
0.7272
2.0598
2.1135
1.9868
2.0007
3.1264
ppm
BRUKER
Current Data Parameters
NAME JKM-341
EXPNO 1
PROCNO 5
F2 - Acquisition Parameters
Date_ 20090426
Time_ 15.01
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 5
DS 0
SWH 6393.852 Hz
FIDRES 0.195125 Hz
AQ 2.5625076 sec
RG 362
DW 78.200 usec
DE 6.00 usec
TE 291.8 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.05 usec
PL1 -3.00 dB
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300104 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

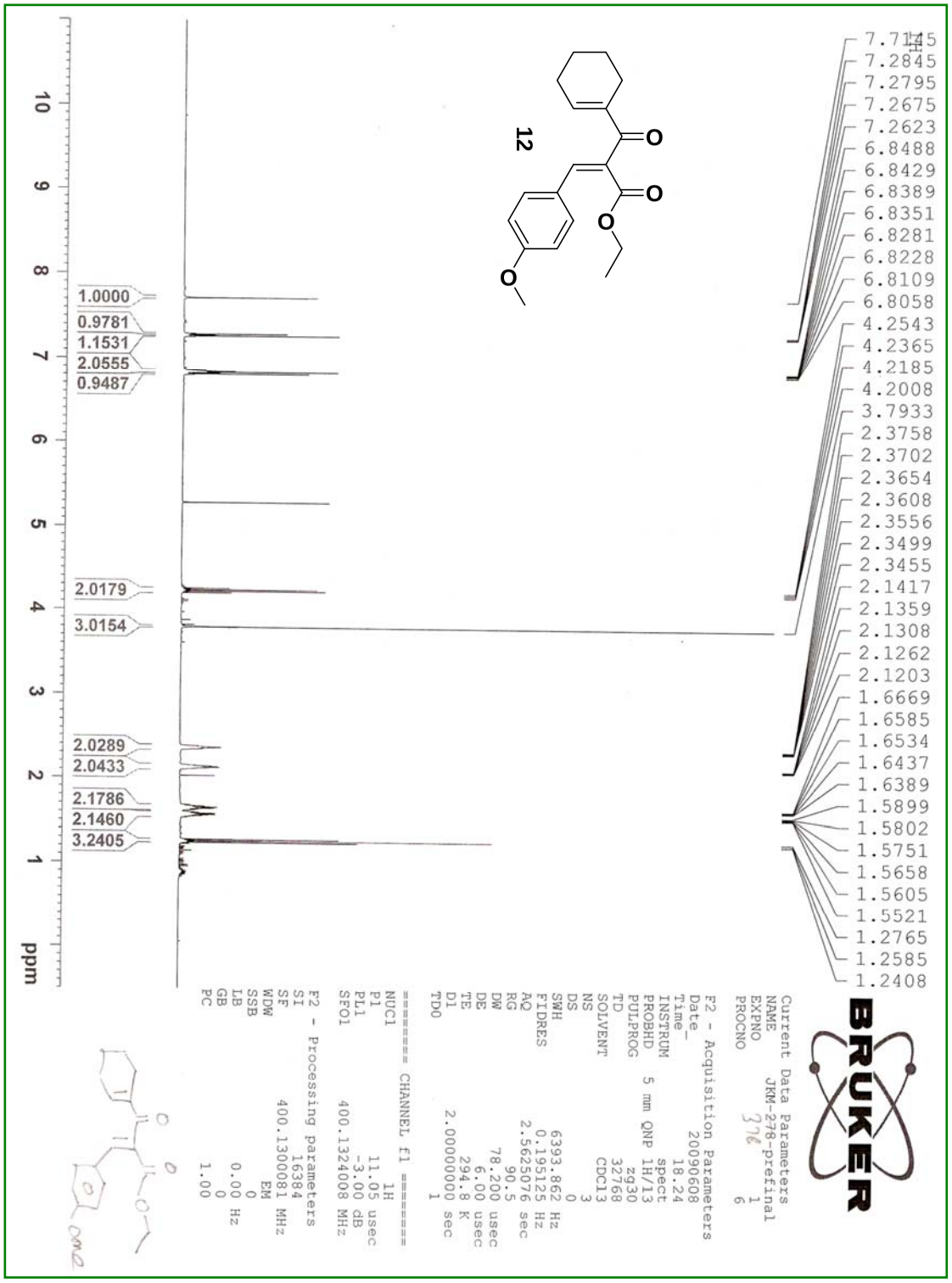

13C
190.70
165.07
151.36
143.16
133.16
130.68
130.50
130.36
128.92
116.32
66.67
61.68
21.46
21.18
14.35
10
O
O
O
O
BRUKER
Current Data Parameters
NAME JKM-341-C13
EXPNO 1
PROCNO 5
F2 - Acquisition Parameters
Date_ 20090426
Time_ 19.39
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 4810
DS 0
SWH 25510.203 Hz
FIDRES 0.389255 Hz
AQ 1.2845556 sec
RG 2050
DW 19.600 usec
DE 7.00 usec
TE 292.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.80 usec
PL1 0.00 dB
SFO1 100.6248425 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -3.00 dB
PL12 14.00 dB
PL13 17.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127551 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00
180
160
140
120
100
80
60
40
20
ppm

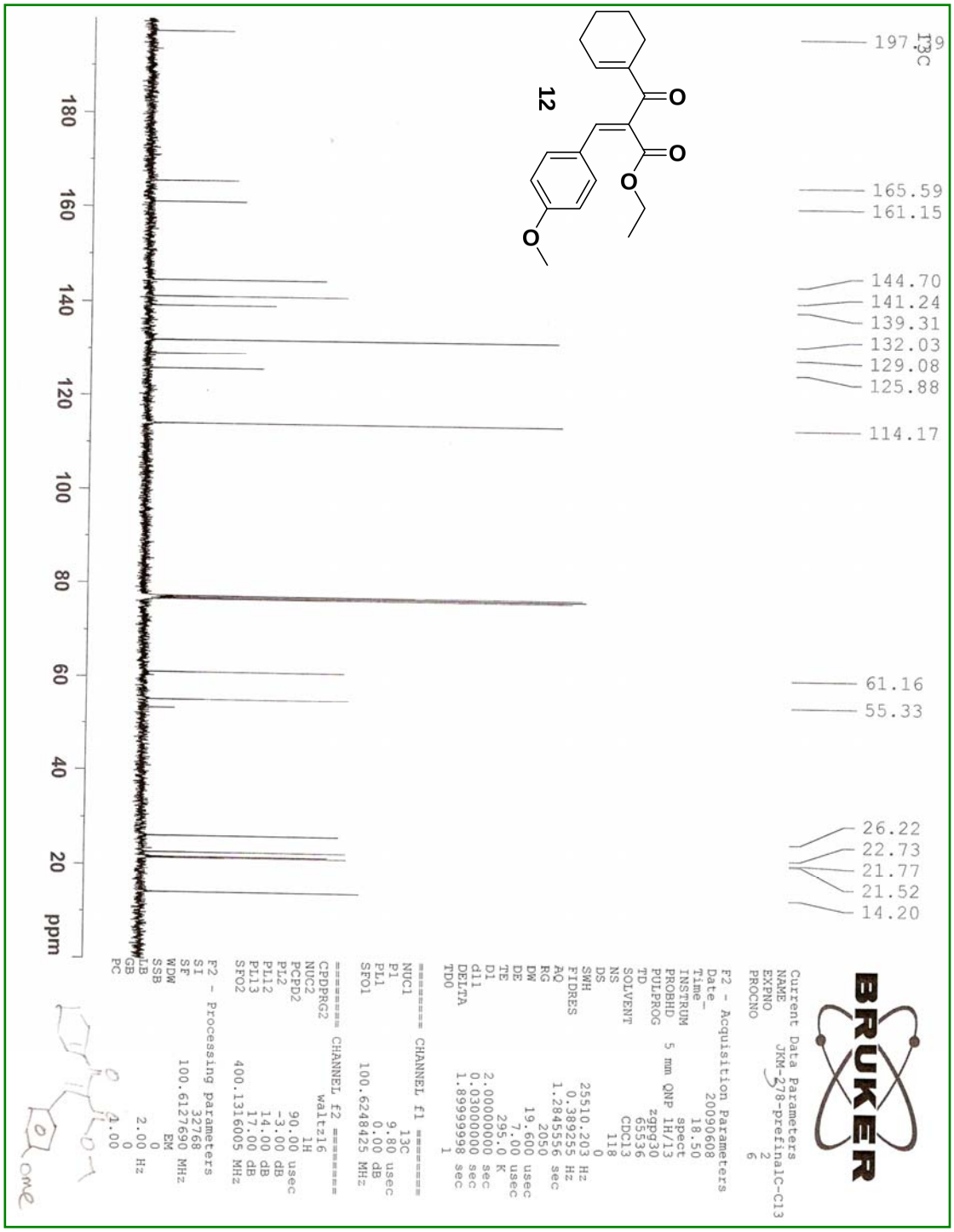


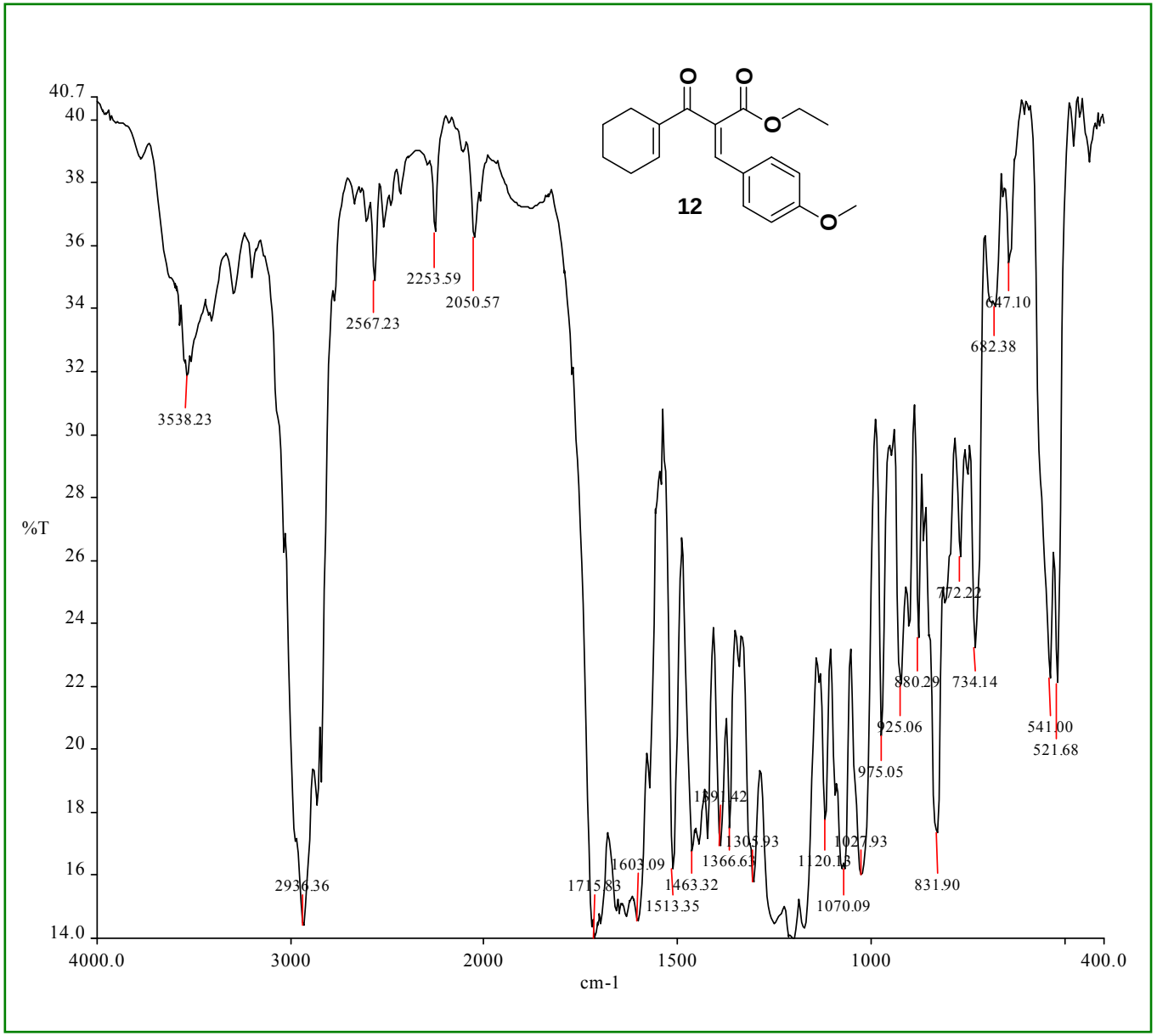

1H
11
O
O
O
O
O
O
7.7540
7.3542
7.3322
6.8538
6.8316
6.0334
6.0228
6.0121
4.2838
4.2660
4.2481
4.2303
4.1223
4.1094
4.0967
3.8143
2.2205
2.2014
2.1743
2.1713
2.1586
2.1476
2.1319
1.8749
1.8590
1.8436
1.8309
1.8173
1.8014
1.5797
1.2969
1.2790
0.9888
2.0000
2.0558
0.9915
2.0926
2.1230
3.1000
2.1539
2.1771
3.3157
ppm
BRUKER
Current Data Parameters
NAME JKM-350
EXPNO 2
PROCNO 5
F2 - Acquisition Parameters
Date_ 20090504
Time_ 14.27
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 3
DS 0
SWH 6393.862 Hz
FIDRES 0.195125 Hz
AQ 2.5625076 sec
RG 456
DW 78.200 usec
DE 6.00 usec
TE 289.4 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.05 usec
PL1 -3.00 dB
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

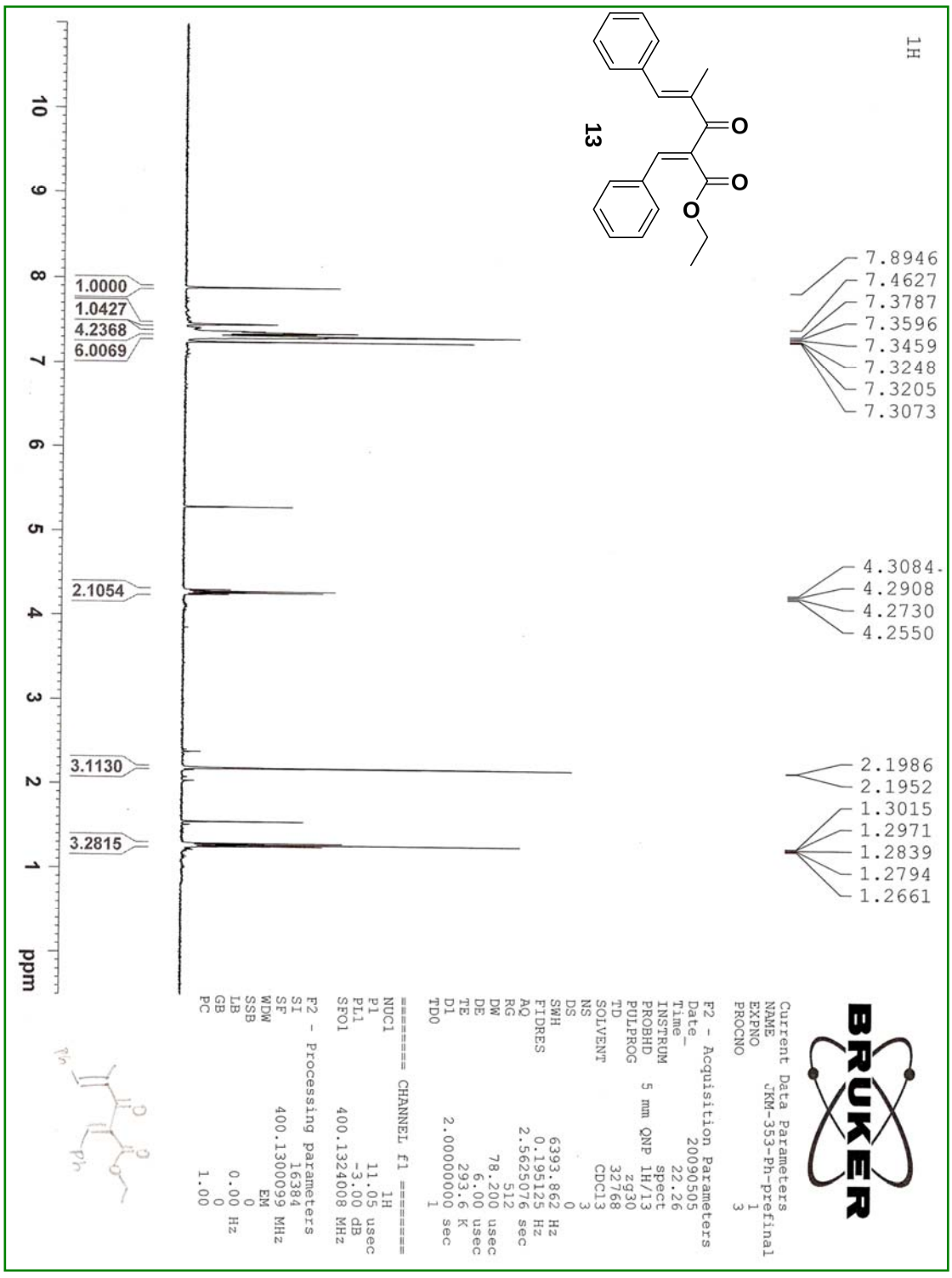

13C
191.11
165.19
161.37
151.24
142.68
132.31
127.71
125.52
114.22
66.53
61.30
55.34
21.31
21.03
14.21
11
O
O
O
O
O
O
180
160
140
120
100
80
60
40
20
ppm
BRUKER
Current Data Parameters
NAME JKM-35@OMe-C13
EXPNO 1
PROCNO 5
F2 - Acquisition Parameters
Date_ 20090504
Time_ 21.46
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 200
DS 0
SWH 25510.203 Hz
FIDRES 0.389255 Hz
AQ 1.2845556 sec
RG 2050
DW 19.600 usec
DE 7.00 usec
TE 290.3 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
========== CHANNEL f1 ==========
NUC1 13C
P1 9.80 usec
PL1 0.00 dB
SFO1 100.6248425 MHz
========== CHANNEL f2 ==========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -3.00 dB
PL12 14.00 dB
PL13 17.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00

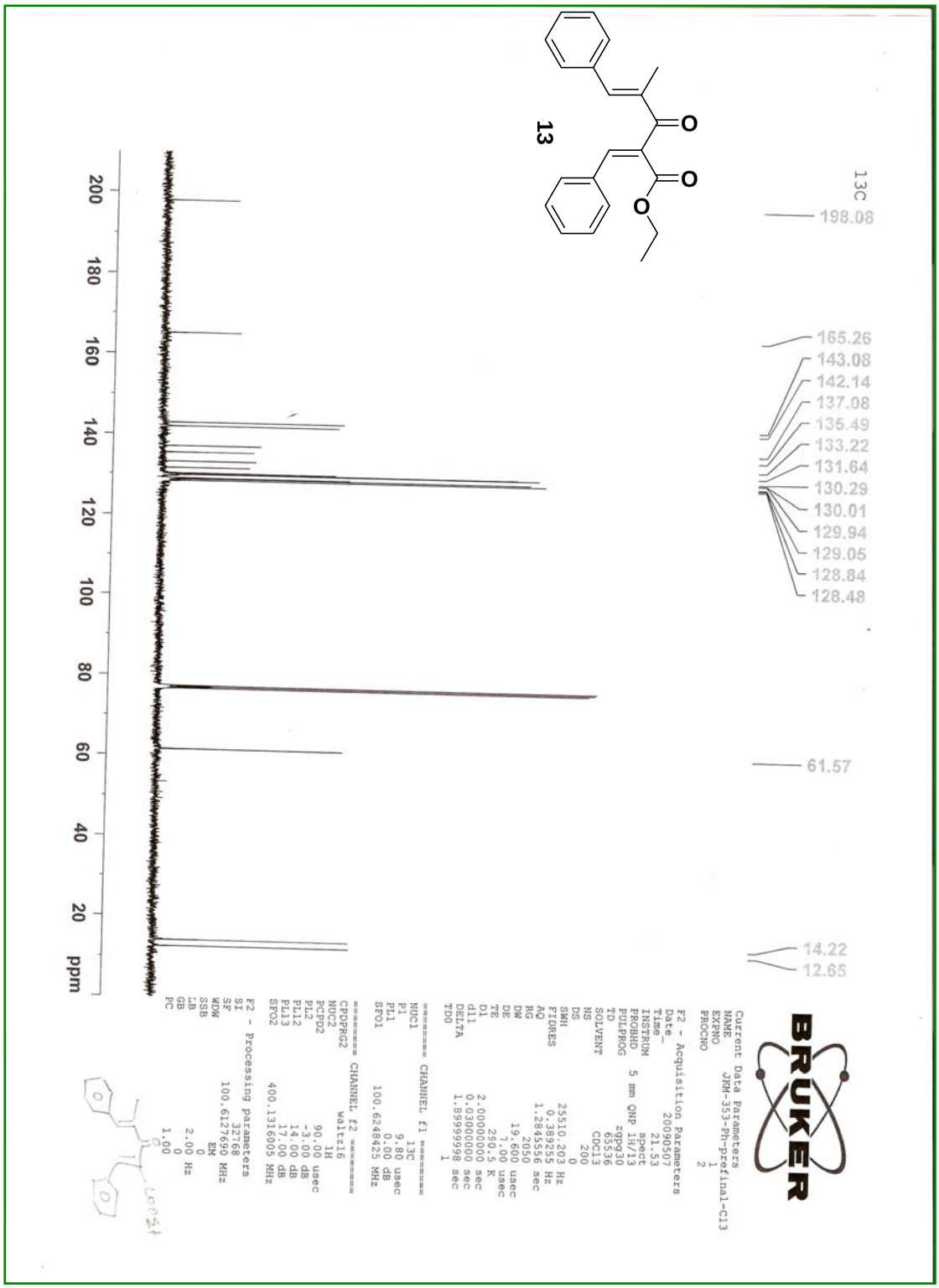

69.8
68
66
64
62
60
58
56
54
52
50
48
46
44
42
40
38
37.0
%T
4000.0
3000
2000
1500
1000
400.0
cm-1
3366.43
2935.88
1714.68
1670.91
1625.23
1602.05
1513.66
1463.84
1425.20
1366.11
1304.40
1250.34
1200.35
1175.18
1099.46
1053.61
1024.20
914.00
831.63
767.56
542.10
O
O
O
O
11

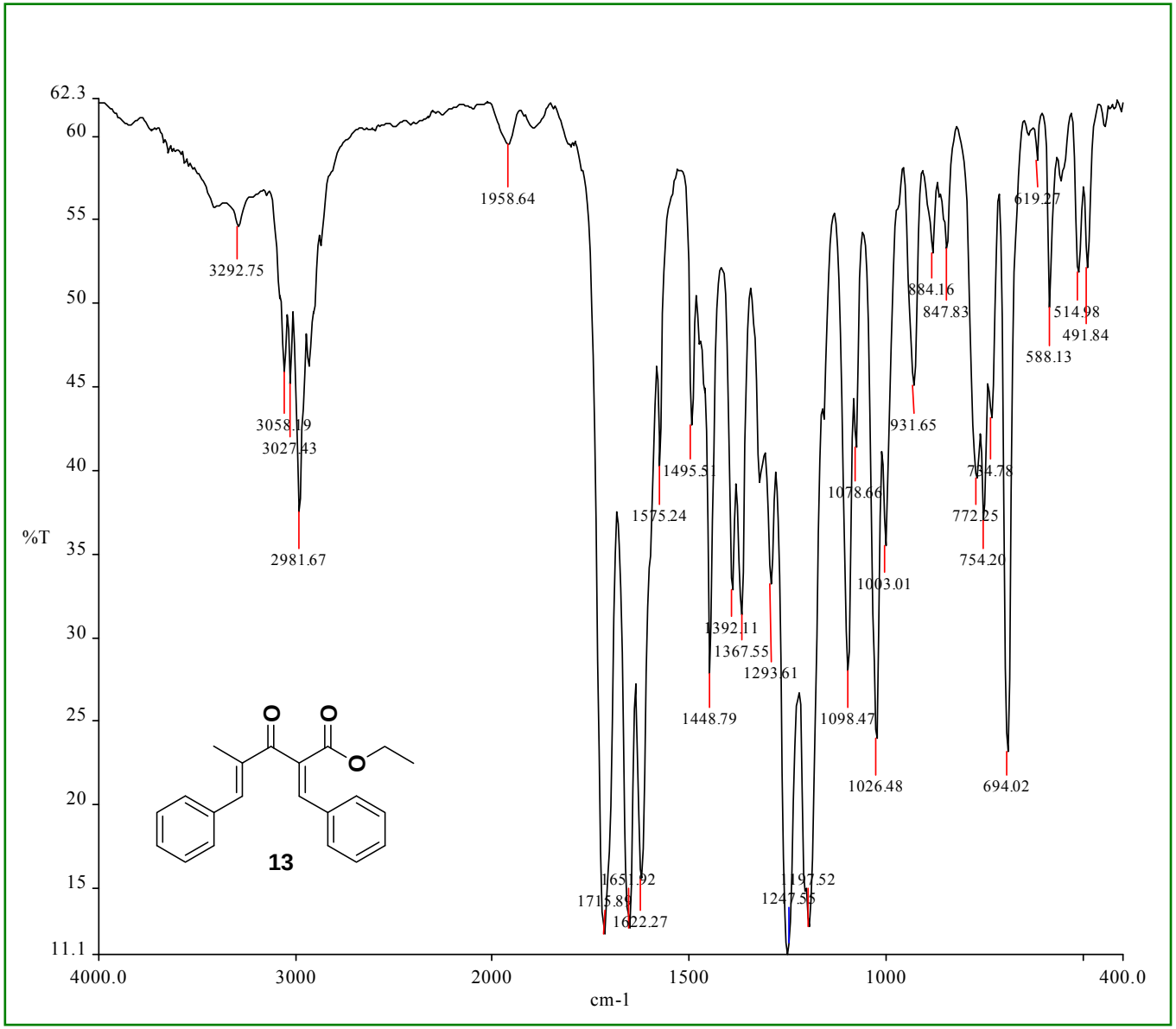

12
1.0000
0.9781
1.1531
2.0555
0.9487
2.0179
3.0154
2.0289
2.0433
2.1786
2.1460
3.2405
ppm
7.7145
7.2845
7.2795
7.2675
7.2623
6.8488
6.8429
6.8389
6.8351
6.8281
6.8228
6.8109
6.8058
4.2543
4.2365
4.2185
4.2008
3.7933
2.3758
2.3702
2.3654
2.3608
2.3556
2.3499
2.3455
2.1417
2.1359
2.1308
2.1262
2.1203
1.6669
1.6585
1.6534
1.6437
1.6389
1.5899
1.5802
1.5751
1.5658
1.5605
1.5521
1.2765
1.2585
1.2408
BRUKER
Current Data Parameters
NAME JKM-278-prefinal
EXPNO 1
PROCNO 6
F2 - Acquisition Parameters
Date_ 20090608
Time_ 18.24
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 3
DS 0
SWH 6393.862 Hz
FIDRES 0.195125 Hz
AQ 2.5625076 sec
RG 90.5
DW 78.200 usec
DE 6.00 usec
TE 294.8 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.05 usec
PL1 -3.00 dB
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300081 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

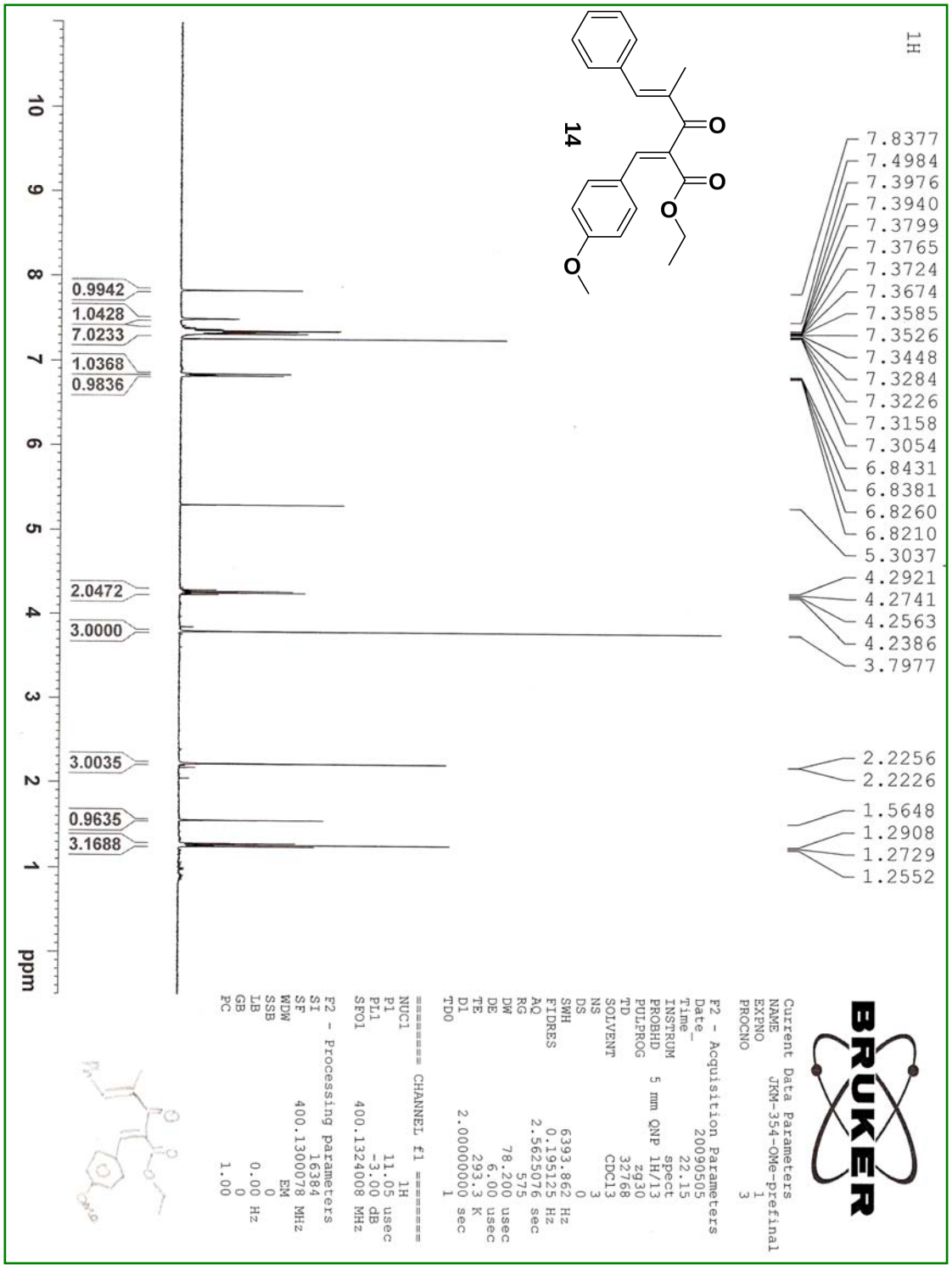

197.39
165.59
161.15
144.70
141.24
139.31
132.03
129.08
125.88
114.17
61.16
55.33
26.22
22.73
21.77
21.52
14.20
12
O
O
O
O
180
160
140
120
100
80
60
40
20
ppm
BRUKER
Current Data Parameters
NAME JKM-276-prefinal-C13
EXPNO 2
PROCNO 6
F2 - Acquisition Parameters
Date_ 20090608
Time_ 18.50
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 118
DS 0
SWH 25510.203 Hz
FIDRES 0.389255 Hz
AQ 1.2845556 sec
RG 2050
DW 19.600 usec
DE 7.00 usec
TE 295.0 K
D1 2.00000000 sec
D11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.80 usec
PL1 0.00 dB
SFO1 100.6248425 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -3.00 dB
PL12 14.00 dB
PL13 17.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 4.00
O
OMe

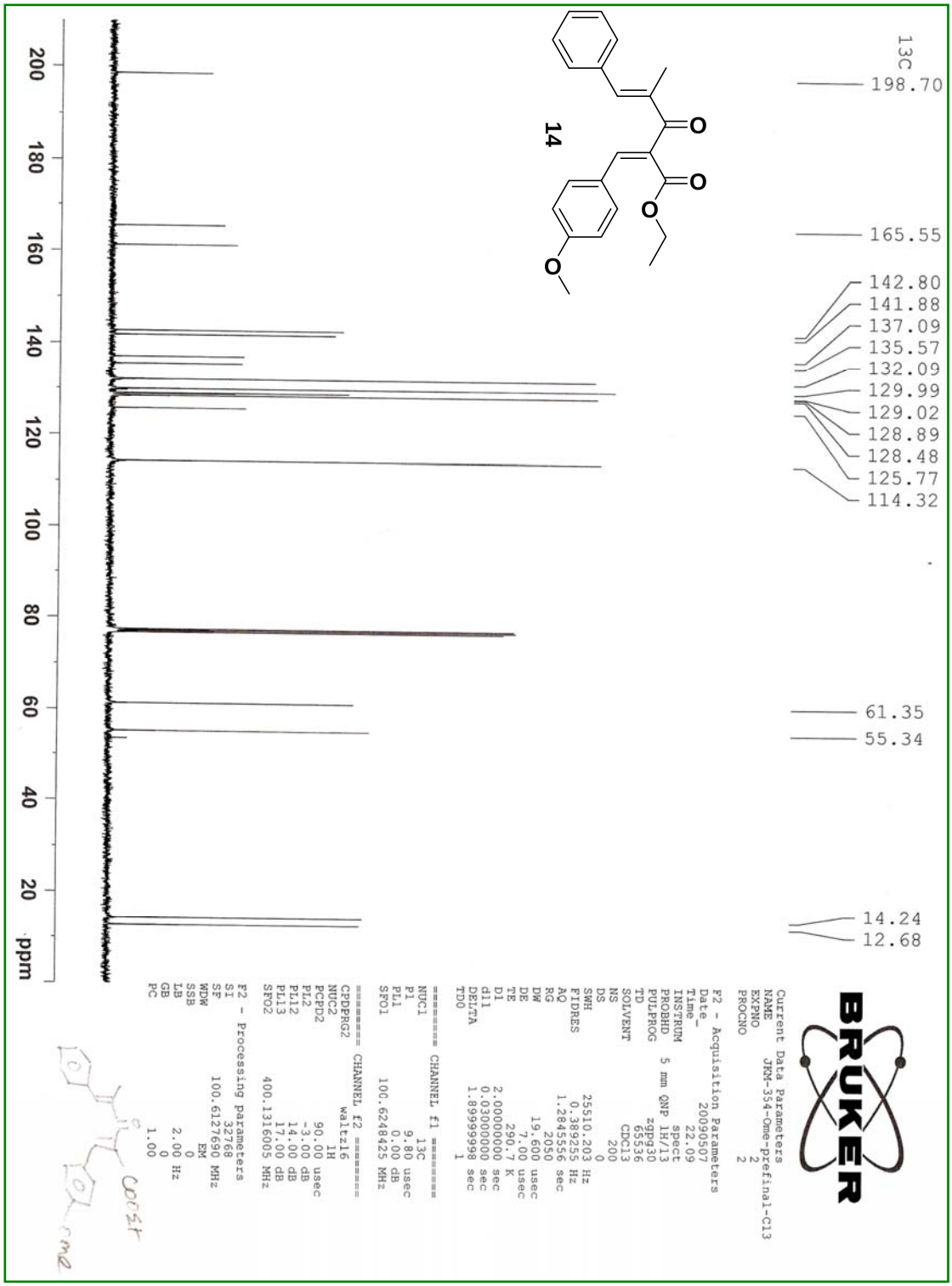


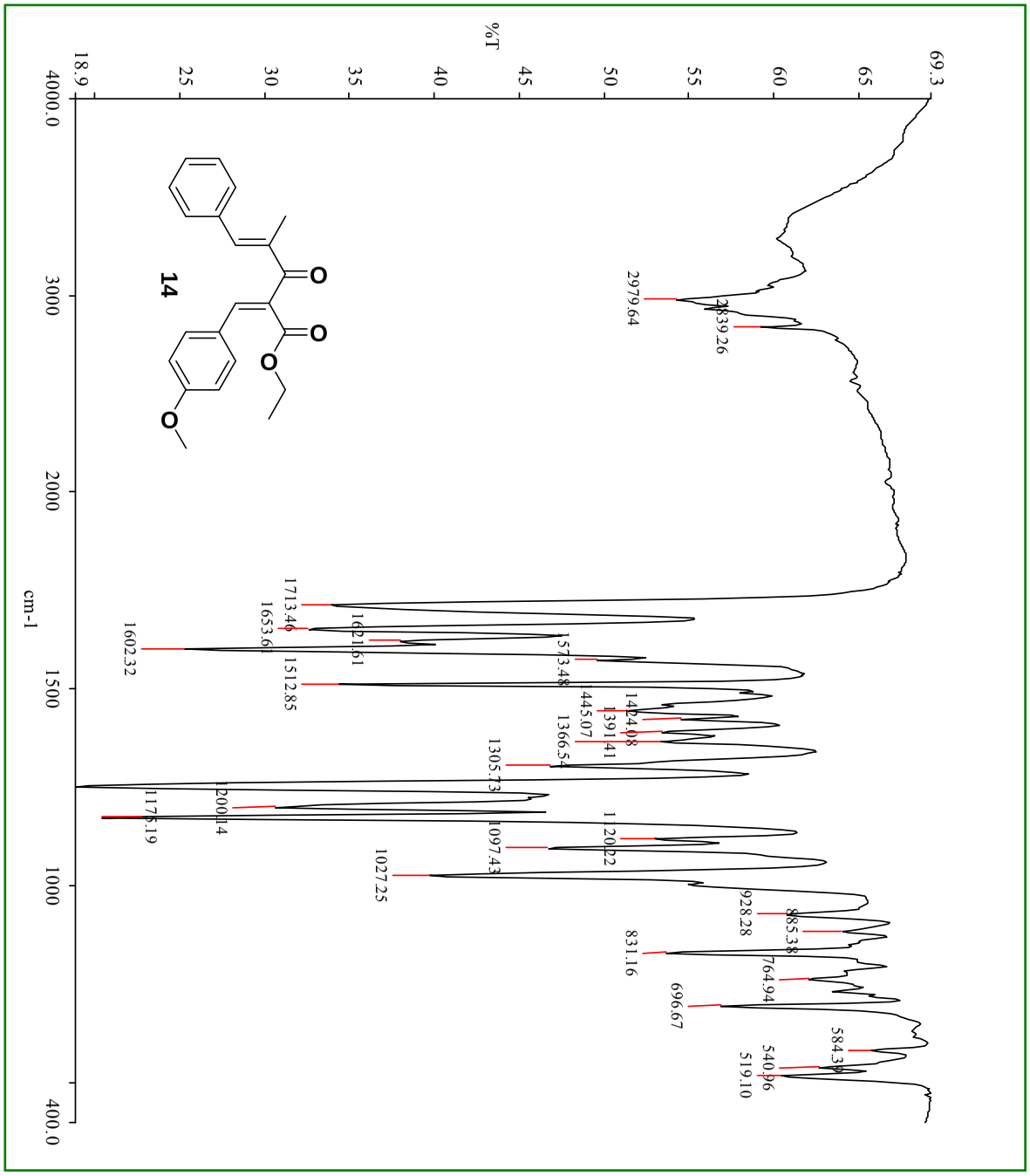

1H
13
7.8946
7.4627
7.3787
7.3596
7.3459
7.3248
7.3205
7.3073
4.3084
4.2908
4.2730
4.2550
2.1986
2.1952
1.3015
1.2971
1.2839
1.2794
1.2661
1.0000
1.0427
4.2368
6.0069
2.1054
3.1130
3.2815
ppm
BRUKER
Current Data Parameters
NAME JKM-353-Ph-prefinal
EXPNO 1
PROCNO 3
F2 - Acquisition Parameters
Date_ 20090505
Time_ 22.26
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 3
DS 0
SWH 6393.862 Hz
FIDRES 0.195125 Hz
AQ 2.5625076 sec
RG 512
DW 78.200 usec
DE 6.00 usec
TE 293.6 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.05 usec
PL1 -3.00 dB
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

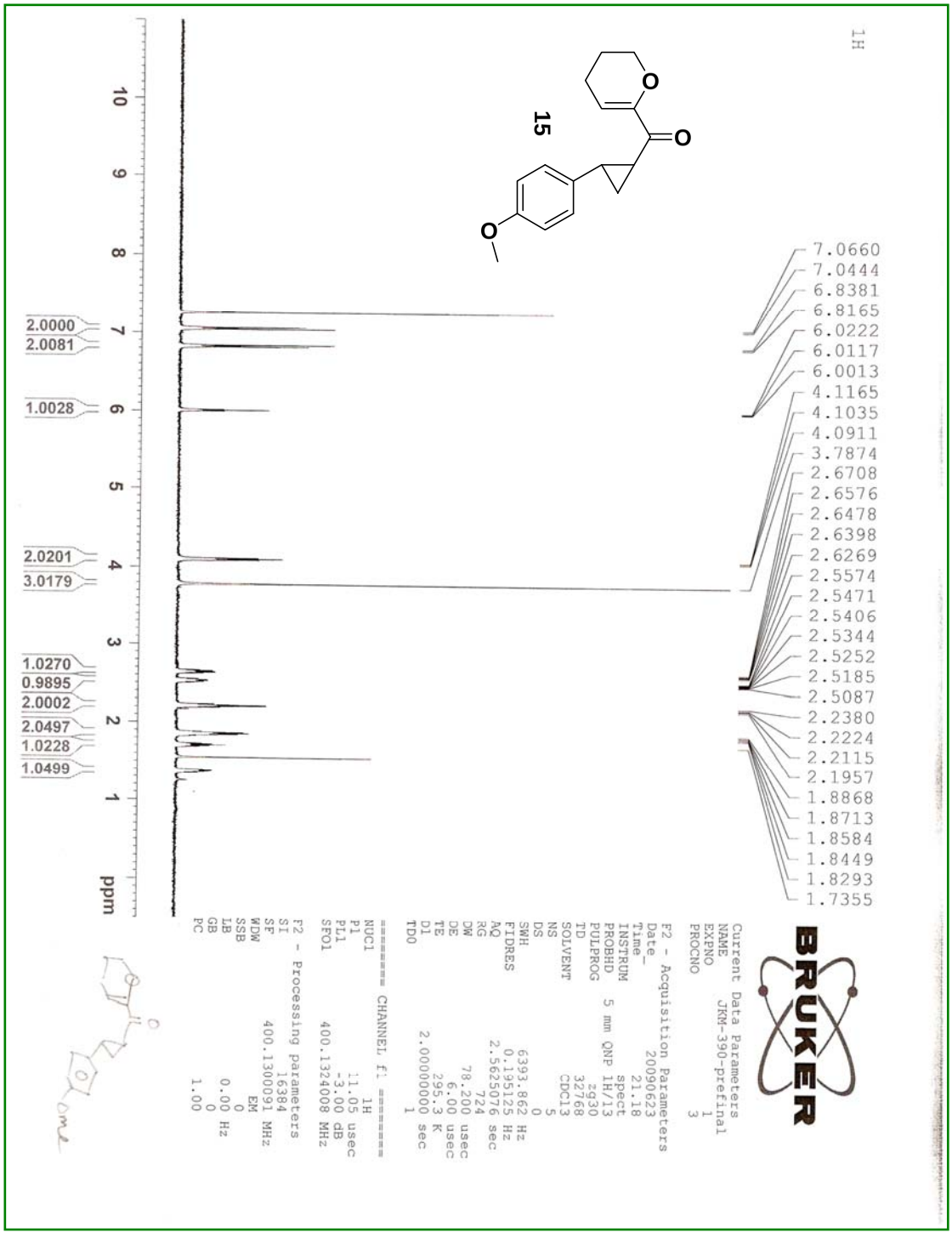

13
O
O
O
13C
198.08
165.26
143.08
142.14
137.08
135.49
133.22
131.64
130.29
130.01
129.94
129.05
128.84
128.48
61.57
14.22
12.65
200
180
160
140
120
100
80
60
40
20
ppm
BRUKER
Current Data Parameters
NAME JRM-353-Ph-prefinal-C13
EXPNO 1
PROCNO 2
F2 - Acquisition Parameters
Date_ 20090507
Time 21.53
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 200
DS 0
SWH 25510.203 Hz
FIDRES 0.389255 Hz
AQ 1.2845556 sec
RG 2050
DW 19.600 usec
DE 7.00 usec
TE 290.5 K
D1 2.00000000 sec
D11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13c
P1 9.80 usec
PL1 0.00 dB
SFO1 100.6248425 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -3.00 dB
PL12 17.00 dB
PL13 14.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00

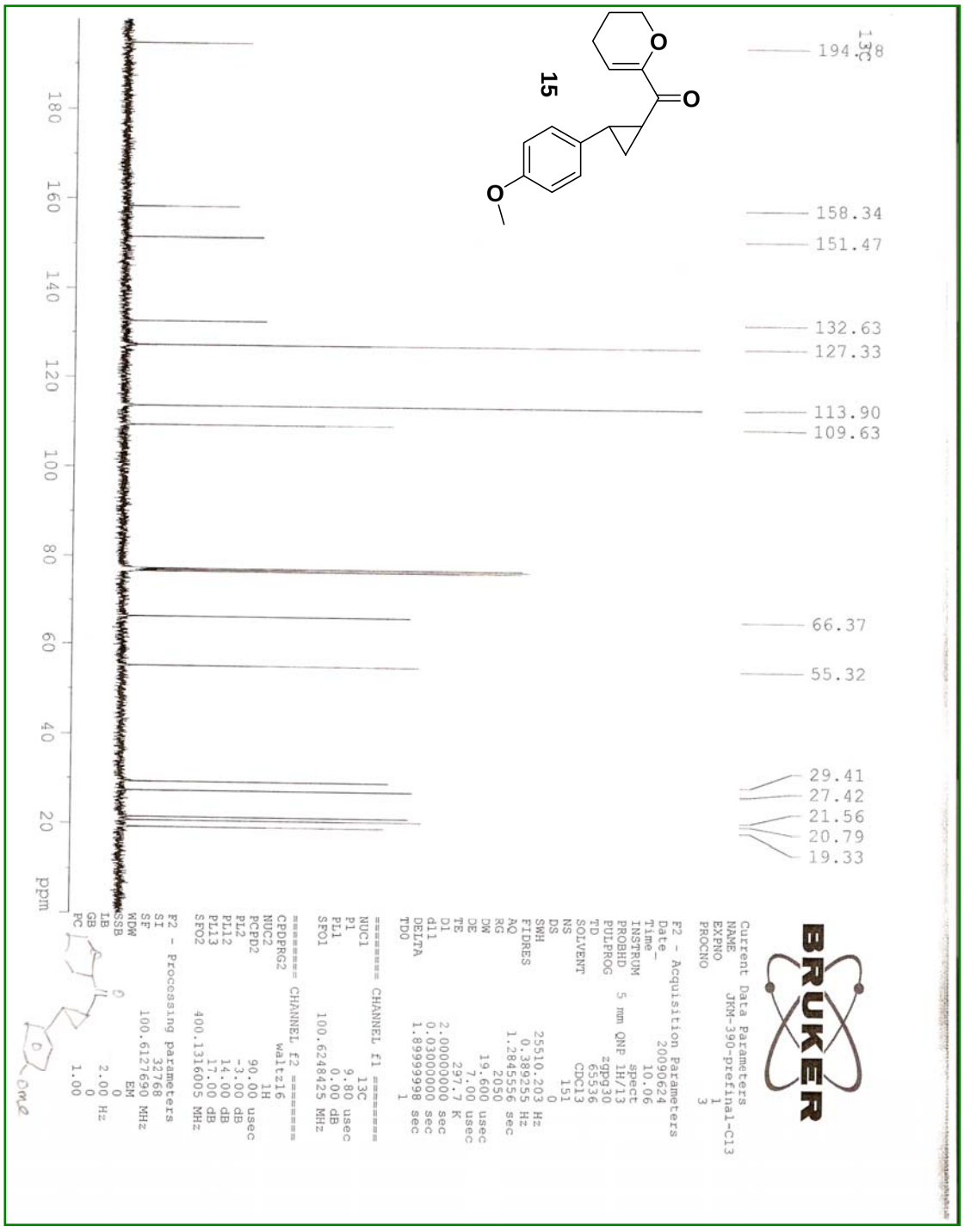


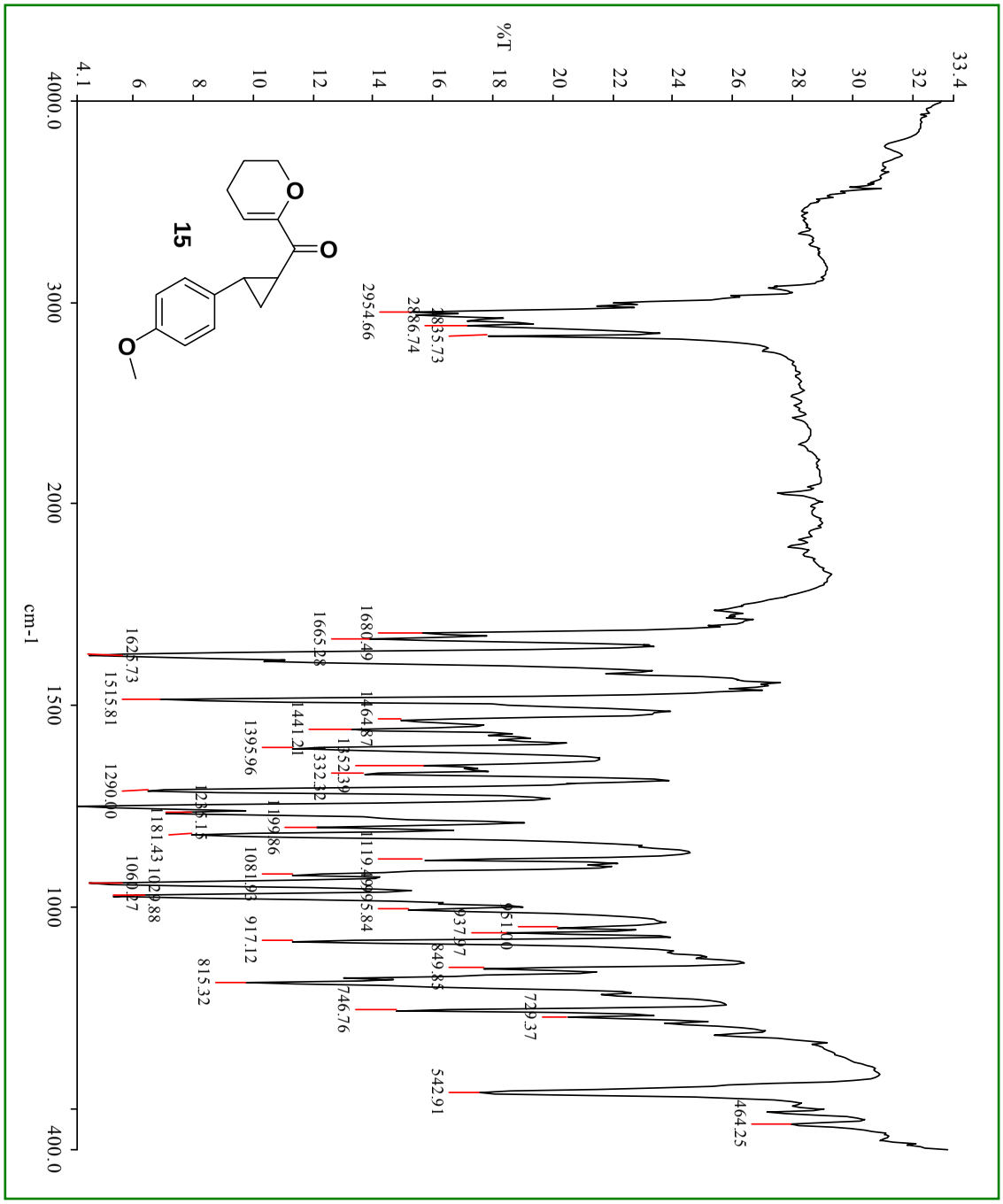

1H
14
7.8377
7.4984
7.3976
7.3940
7.3799
7.3765
7.3724
7.3674
7.3585
7.3526
7.3448
7.3284
7.3226
7.3158
7.3054
6.8431
6.8381
6.8260
6.8210
5.3037
4.2921
4.2741
4.2563
4.2386
3.7977
2.2256
2.2226
1.5648
1.2908
1.2729
1.2552
0.9942
1.0428
7.0233
1.0368
0.9836
2.0472
3.0000
3.0035
0.9635
3.1688
ppm
BRUKER
Current Data Parameters
NAME JKM-354-OMe-prefinal
EXPNO 1
PROCNO 3
F2 - Acquisition Parameters
Date_ 20090505
Time_ 22.15
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 3
DS 0
SWH 6393.862 Hz
FIDRES 0.195125 Hz
AQ 2.5625076 sec
RG 575
DW 78.200 usec
DE 6.00 usec
TE 293.3 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.05 usec
PL1 -3.00 dB
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300078 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

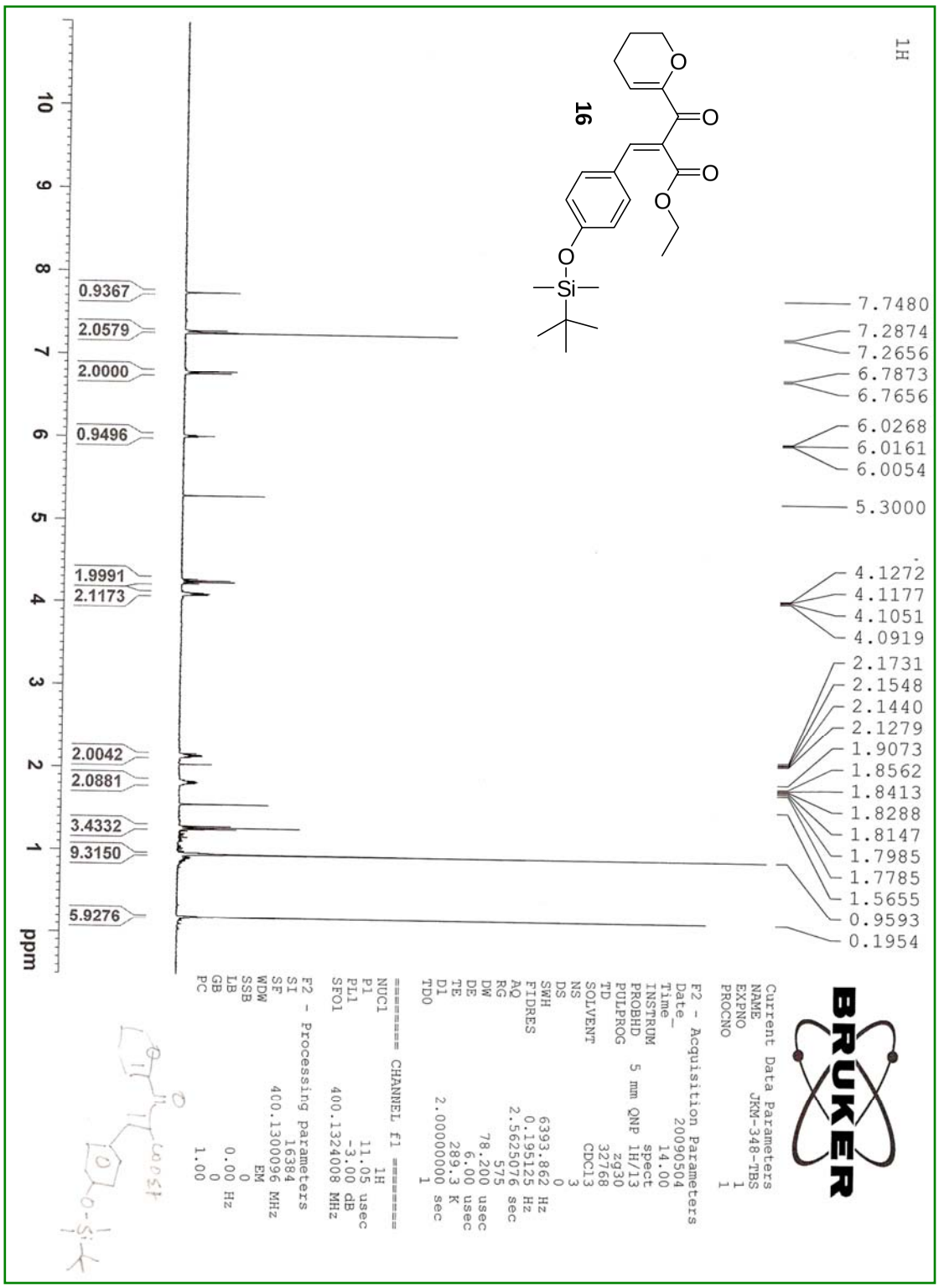

13C
198.70
165.55
142.80
141.88
137.09
135.57
132.09
129.99
129.02
128.89
128.48
125.77
114.32
61.35
55.34
14.24
12.68
14
O
O
O
O
200
180
160
140
120
100
80
60
40
20
ppm
BRUKER
Current Data Parameters
NAME JKM-354-Ome-prefinal-C13
EXPNO 2
PROCNO 2
F2 - Acquisition Parameters
Date_ 20090507
Time 22.09
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 200
DS 0
SWH 25510.203 Hz
FIDRES 0.389255 Hz
AQ 1.2845556 sec
RG 2050
DW 19.600 usec
DE 7.00 usec
TE 290.7 K
D1 2.00000000 sec
D11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.80 usec
PL1 0.00 dB
SFO1 100.6248425 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -3.00 dB
PL12 14.00 dB
PL13 17.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00

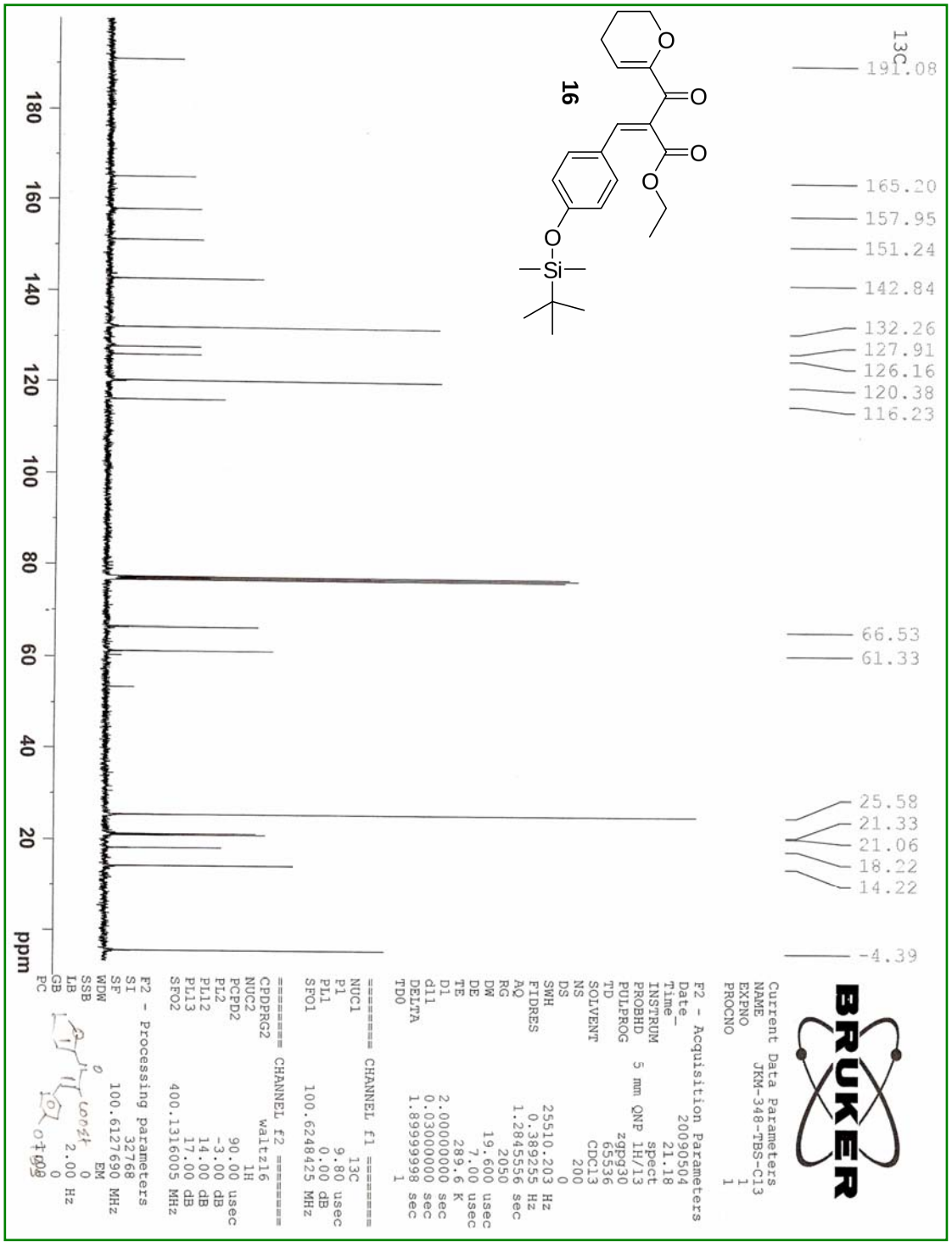

14
%T
cm-1
69.3
65
60
55
50
45
40
35
30
25
18.9
4000.0
3000
2000
1500
1000
400.0
2979.64
2839.26
1713.46
1653.61
1602.32
1512.85
1481.61
1573.48
1445.07
1424.08
1391.41
1366.54
1305.73
1200.14
1178.19
1120.22
1097.43
1027.25
928.28
885.38
831.16
764.94
696.67
584.39
540.96
519.10

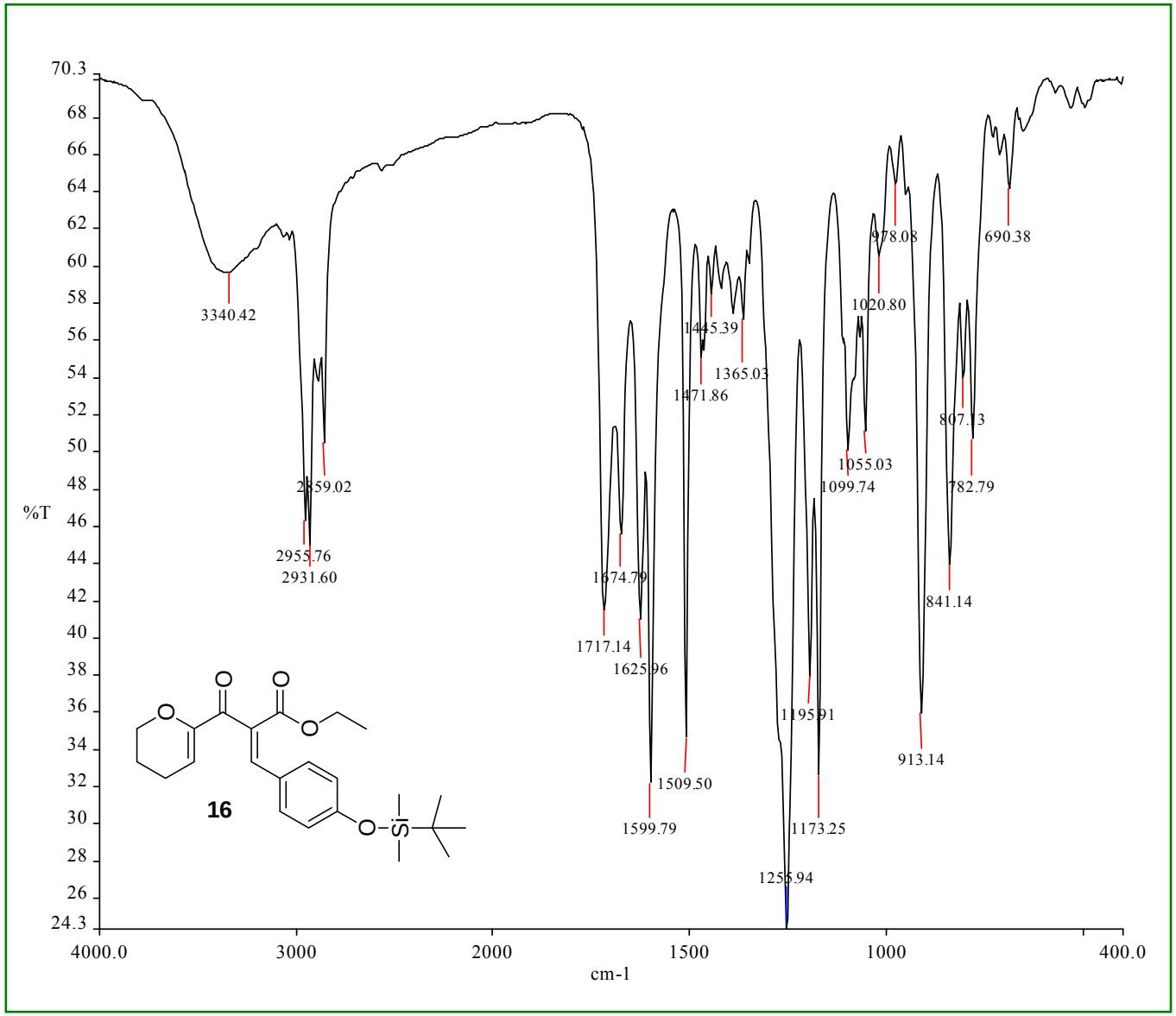

1H
15
O
O
O
7.0660
7.0444
6.8381
6.8165
6.0222
6.0117
6.0013
4.1165
4.1035
4.0911
3.7874
2.6708
2.6576
2.6478
2.6398
2.6269
2.5574
2.5471
2.5406
2.5344
2.5252
2.5185
2.5087
2.2380
2.2224
2.2115
2.1957
1.8868
1.8713
1.8584
1.8449
1.8293
1.7355
2.0000
2.0081
1.0028
2.0201
3.0179
1.0270
0.9895
2.0002
2.0497
1.0228
1.0499
ppm
BRUKER
Current Data Parameters
NAME JKM-390-prefinal
EXPNO 1
PROCNO 3
F2 - Acquisition Parameters
Date_ 20090623
Time_ 21.18
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 5
DS 0
SWH 6393.862 Hz
FIDRES 0.195125 Hz
AQ 2.5625076 sec
RG 724
DW 78.200 usec
DE 6.00 usec
TE 295.3 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.05 usec
PL1 -3.00 dB
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300091 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

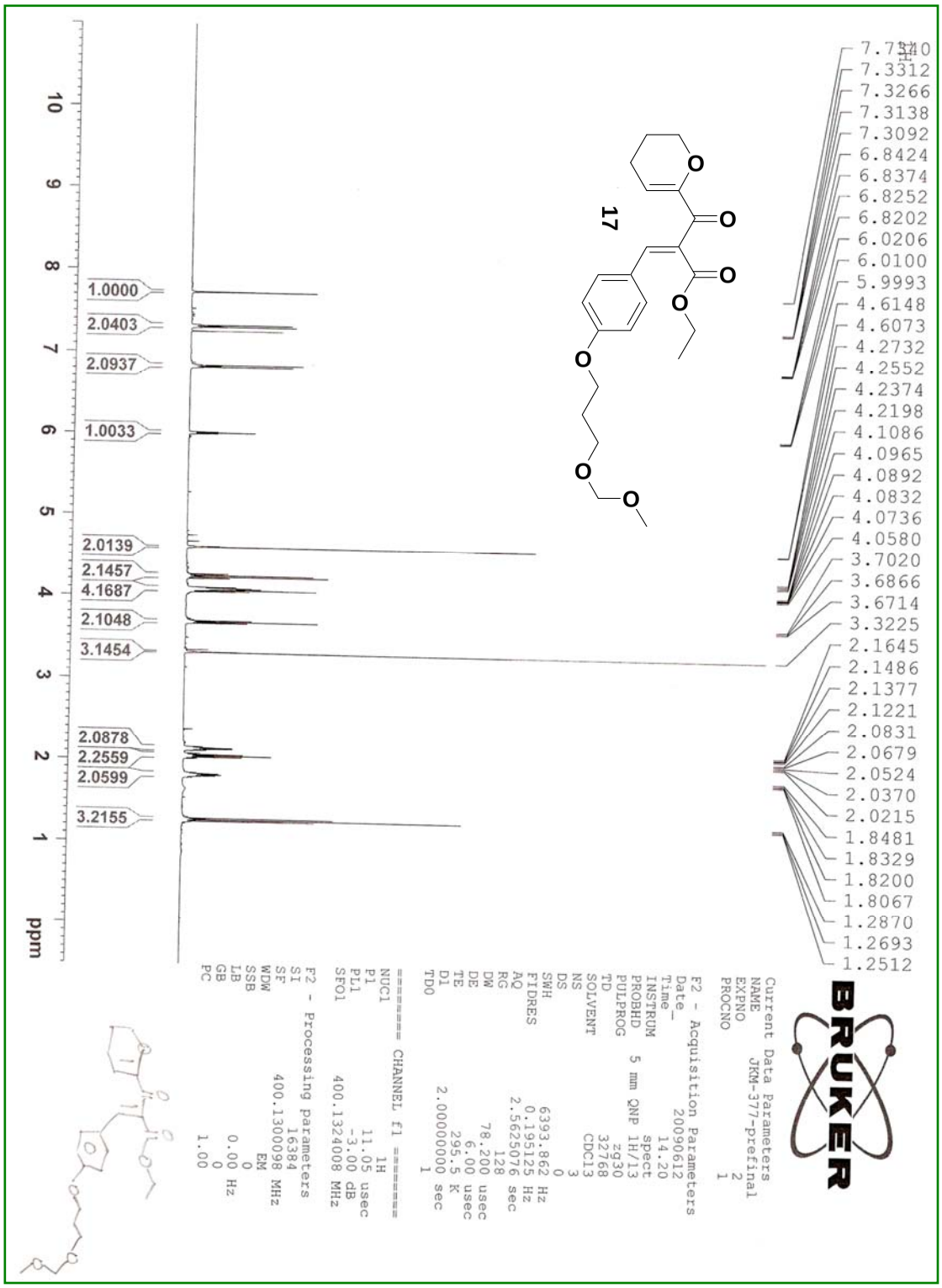

15
O
O
O
194.38
158.34
151.47
132.63
127.33
113.90
109.63
66.37
55.32
29.41
27.42
21.56
20.79
19.33
ppm
BRUKER

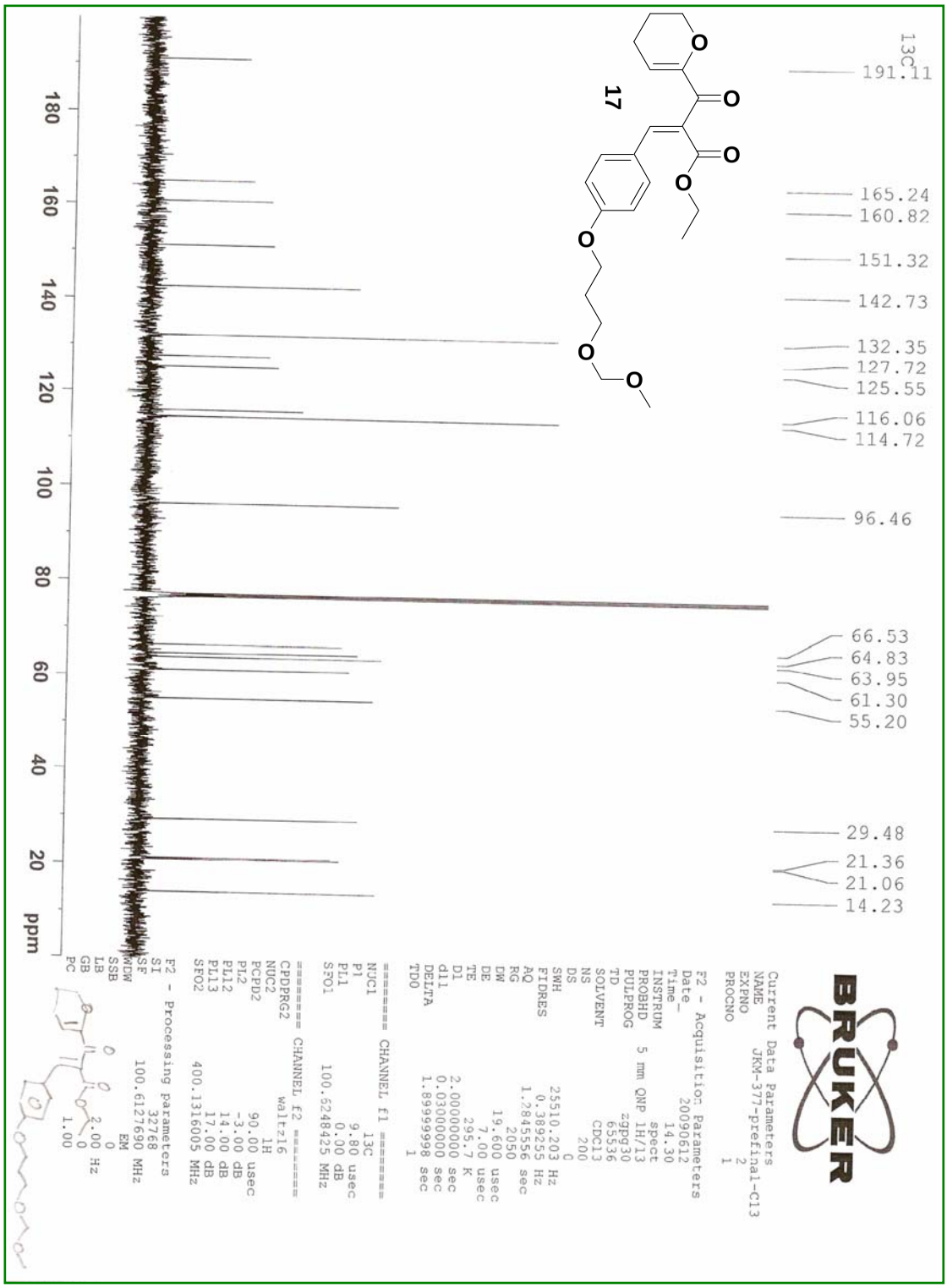

15
%T
cm-1
4000.0
3000
2000
1500
1000
400.0
2954.66
2835.73
2786.74
1680.49
1665.28
1622.73
1515.81
1464.87
1441.21
1395.96
1352.39
1332.32
1290.00
1235.15
1199.86
1181.43
1119.40
1081.93
1060.27
1029.88
995.84
937.97
917.12
851.00
849.85
815.32
746.76
739.37
542.91
464.25

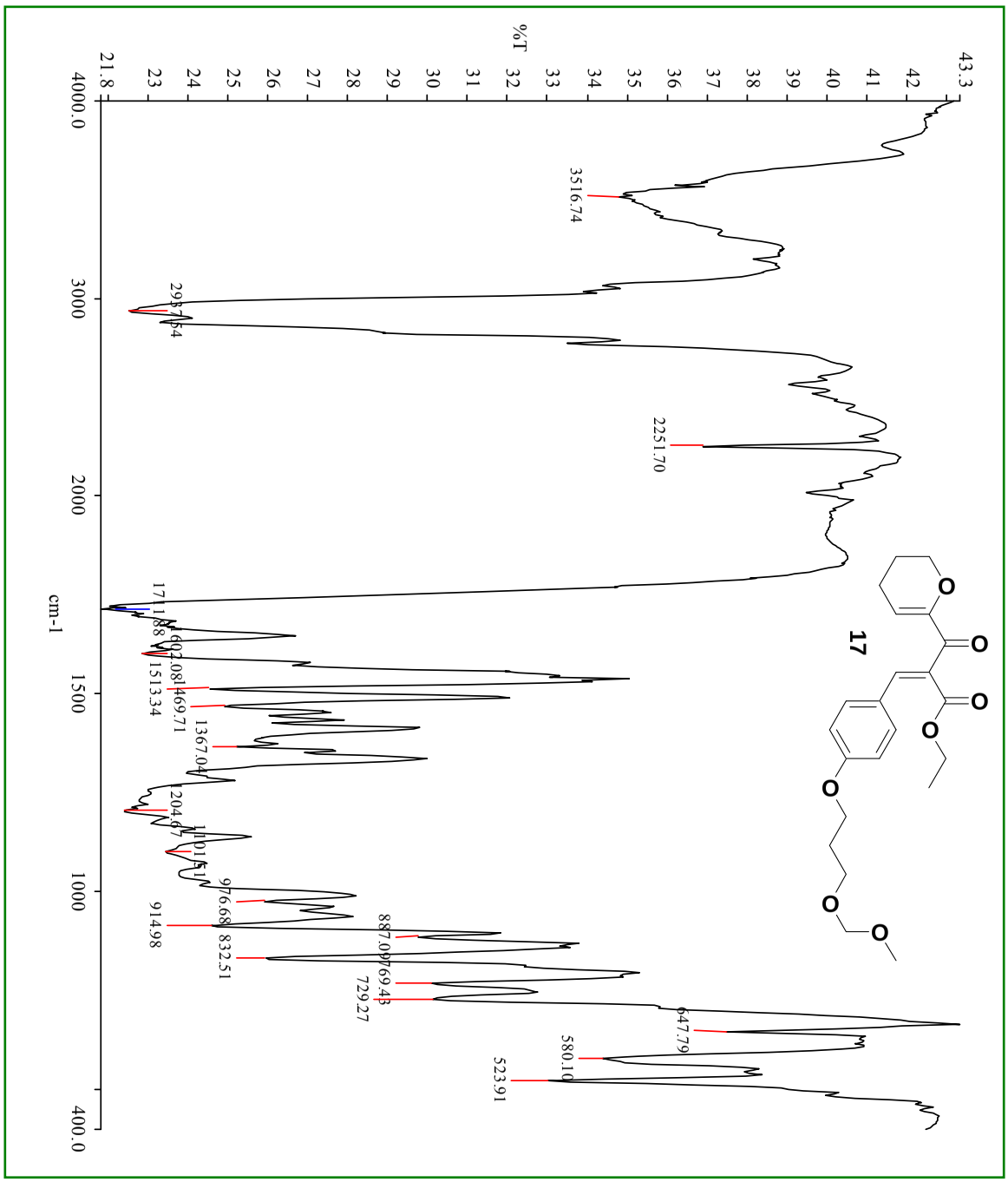

1H
16
O
O
O
O
O
O
Si
7.7480
7.2874
7.2656
6.7873
6.7656
6.0268
6.0161
6.0054
5.3000
4.1272
4.1177
4.1051
4.0919
2.1731
2.1548
2.1440
2.1279
1.9073
1.8562
1.8413
1.8288
1.8147
1.7985
1.7785
1.5655
0.9593
0.1954
0.9367
2.0579
2.0000
0.9496
1.9991
2.1173
2.0042
2.0881
3.4332
9.3150
5.9276
ppm
BRUKER
Current Data Parameters
NAME JKM-348-TBS
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090504
Time 14.00
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 3
DS 0
SWH 6393.862 Hz
FIDRES 0.195125 Hz
AQ 2.5625076 sec
RG 575
DW 78.200 usec
DE 6.00 usec
TE 289.3 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.05 usec
PL1 -3.00 dB
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

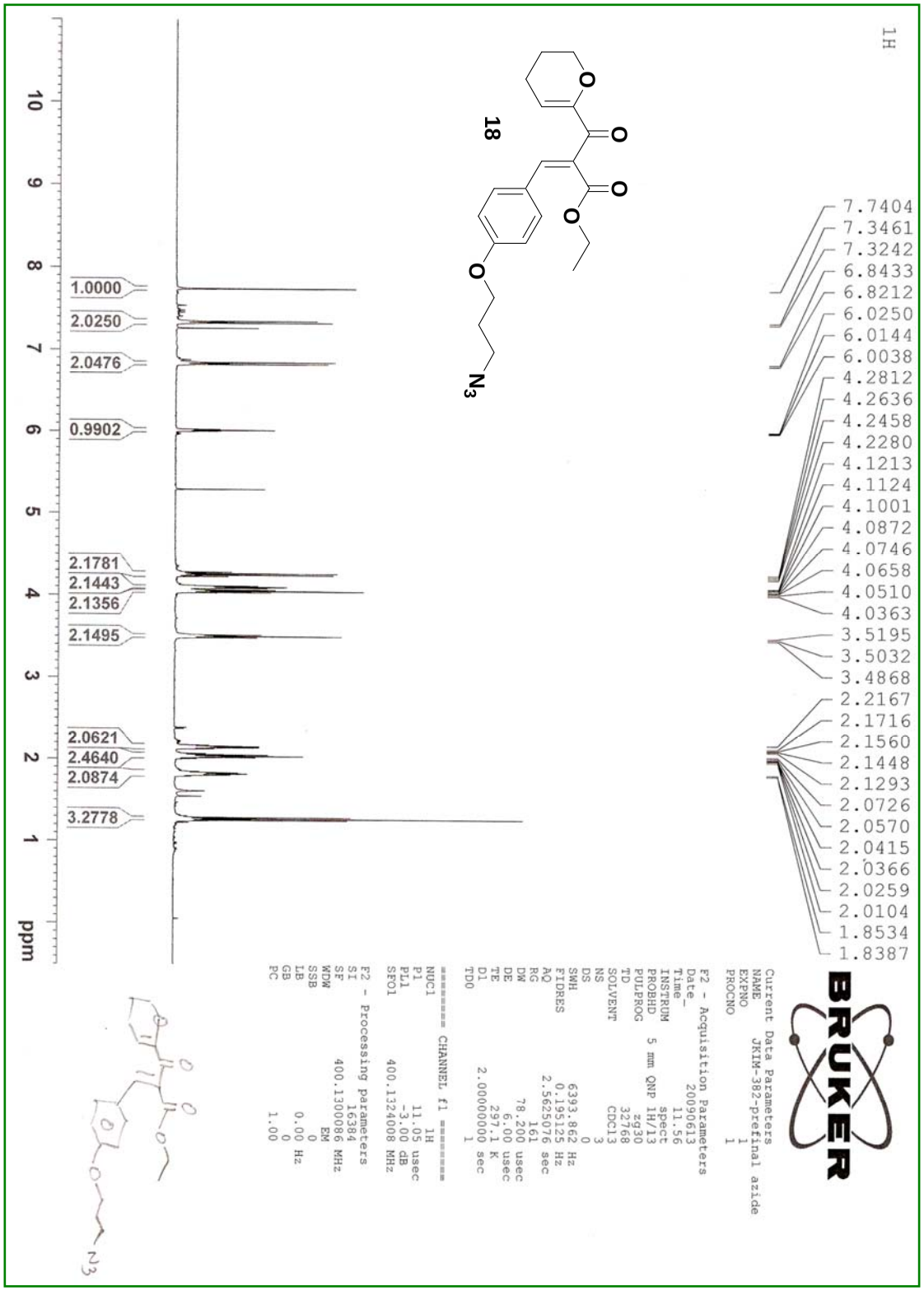

13C
191.08
165.20
157.95
151.24
142.84
132.26
127.91
126.16
120.38
116.23
66.53
61.33
25.58
21.33
21.06
18.22
14.22
-4.39
16
O
O
O
O
O
Si
BRUKER
Current Data Parameters
NAME JKM-348-TBS-C13
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090504
Time_ 21.18
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 200
DS 0
SWH 25510.203 Hz
FIDRES 0.389255 Hz
AQ 1.2845556 sec
RG 2050
DW 19.600 usec
DE 7.00 usec
TE 289.6 K
D1 2.00000000 sec
D11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.80 usec
PL1 0.00 dB
SFO1 100.6248425 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -3.00 dB
PL12 14.00 dB
PL13 17.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC
180
160
140
120
100
80
60
40
20
ppm

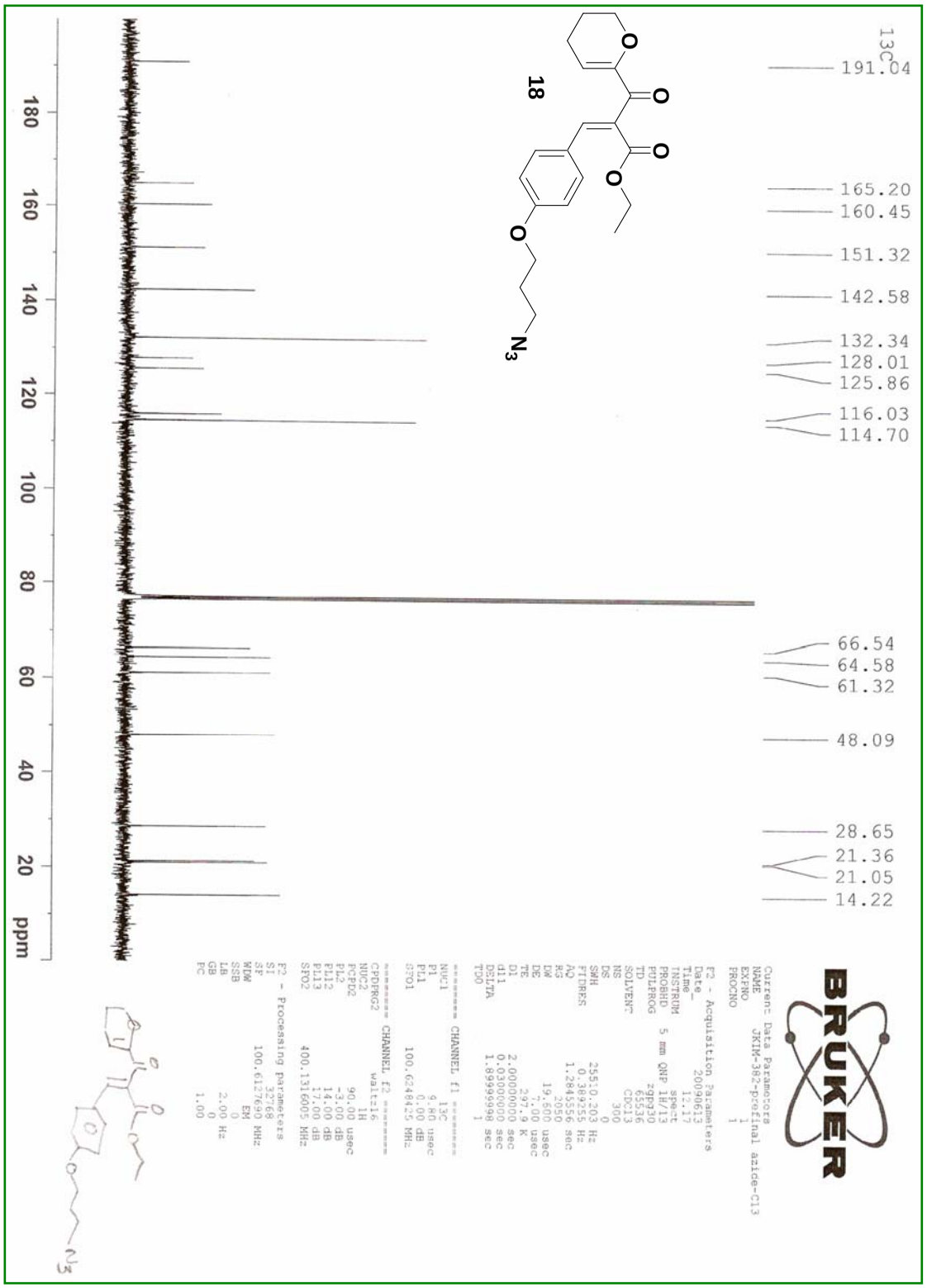


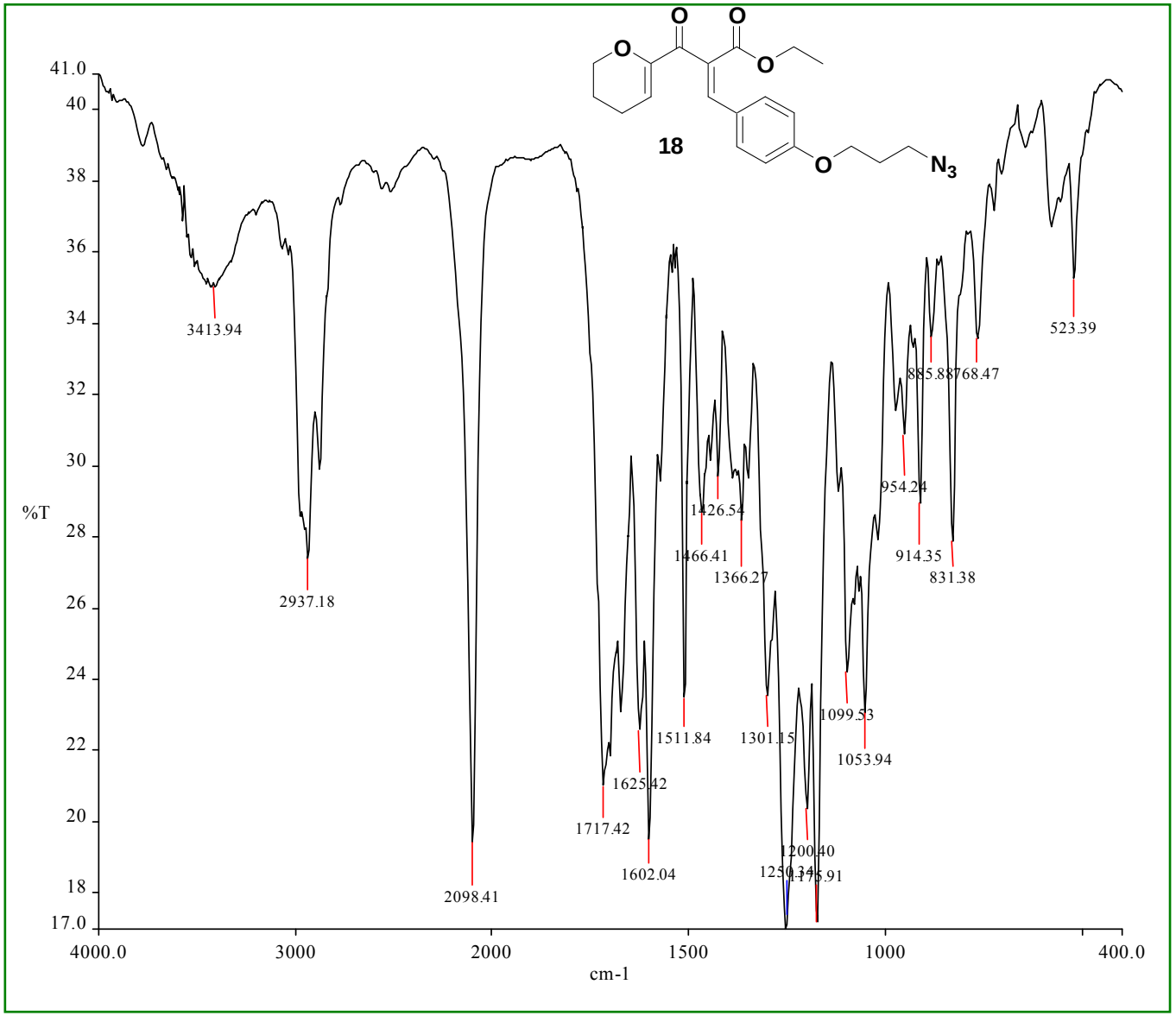

17
1.0000
2.0403
2.0937
1.0033
2.0139
2.1457
4.1687
2.1048
3.1454
2.0878
2.2559
2.0599
3.2155
ppm
7.7340
7.3312
7.3266
7.3138
7.3092
6.8424
6.8374
6.8252
6.8202
6.0206
6.0100
5.9993
4.6148
4.6073
4.2732
4.2552
4.2374
4.2198
4.1086
4.0965
4.0892
4.0832
4.0736
4.0580
3.7020
3.6866
3.6714
3.3225
2.1645
2.1486
2.1377
2.1221
2.0831
2.0679
2.0524
2.0370
2.0215
1.8481
1.8329
1.8200
1.8067
1.2870
1.2693
1.2512
Current Data Parameters
NAME JKM-377-prefinal
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090612
Time_ 14.20
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 3
DS 0
SWH 6393.862 Hz
FIDRES 0.195125 Hz
AQ 2.5625076 sec
RG 128
DW 78.200 usec
DE 6.00 usec
TE 295.5 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.05 usec
PL1 -3.00 dB
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300098 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
BRUKER

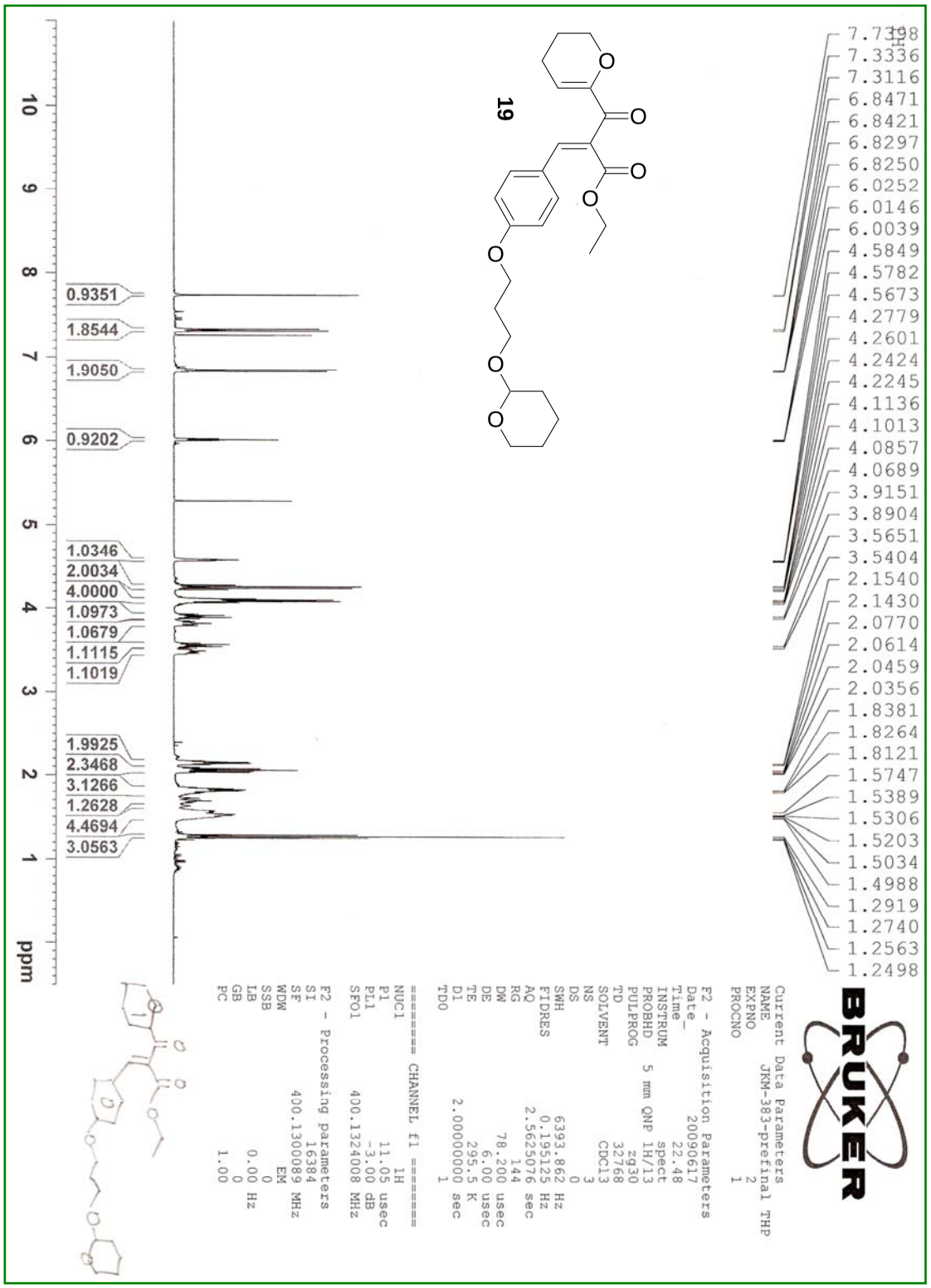

13C
191.11
165.24
160.82
151.32
142.73
132.35
127.72
125.55
116.06
114.72
96.46
66.53
64.83
63.95
61.30
55.20
29.48
21.36
21.06
14.23
17
ppm
Current Data Parameters
NAME JKM-377-prefinal-C13
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090612
Time_ 14.30
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 200
DS 0
SWH 25510.203 Hz
FIDRES 0.389255 Hz
AQ 1.2845556 sec
RG 2050
DW 19.600 usec
DE 7.00 usec
TE 295.7 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.80 usec
PL1 0.00 dB
SFO1 100.6248425 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -3.00 dB
PL12 14.00 dB
PL13 17.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00

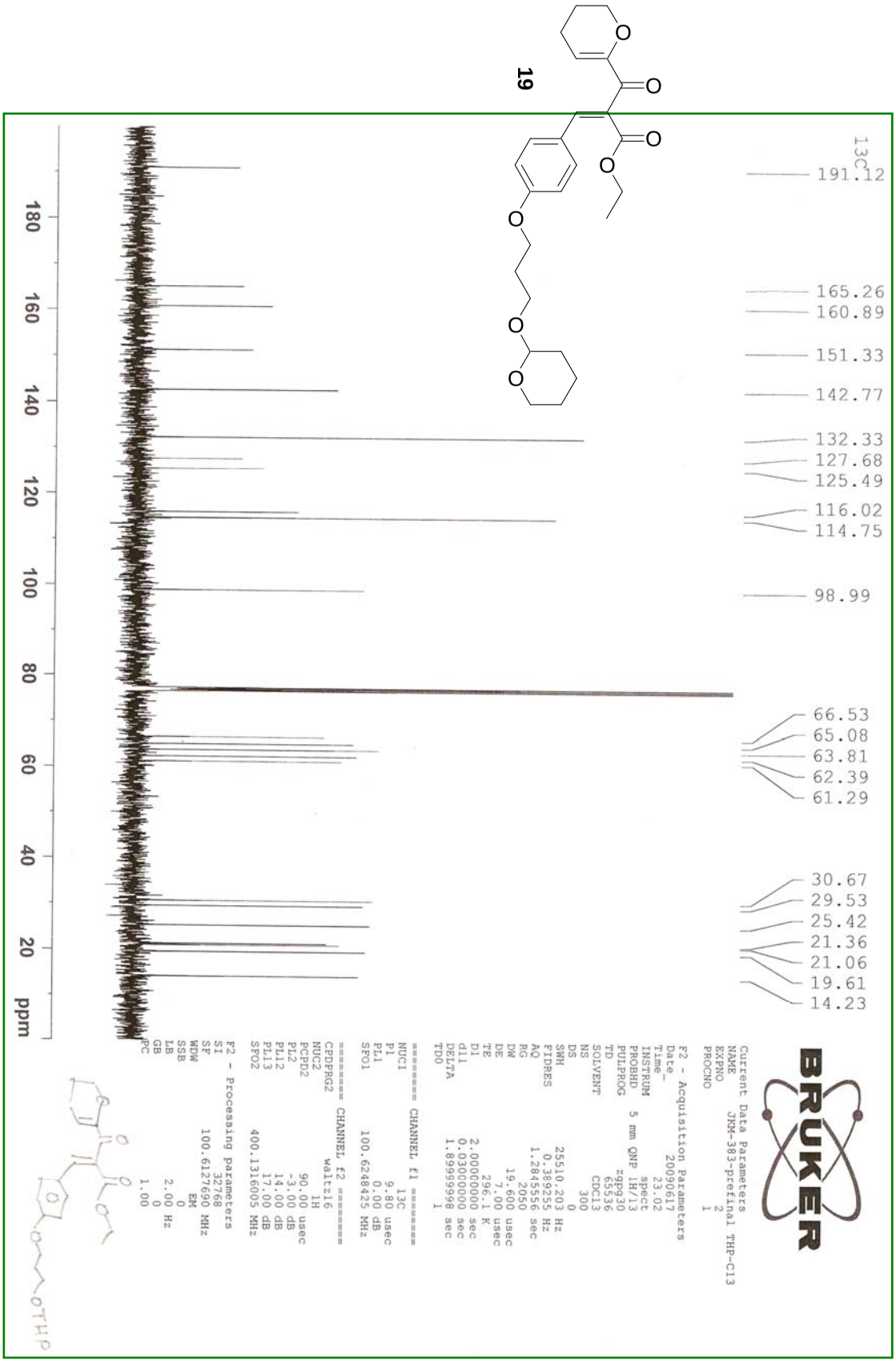

%T
cm-1
3516.74
2937.54
2251.70
1711.88
1602.08
1513.34
1469.71
1367.04
1204.67
1101.51
976.68
914.98
887.09
832.51
769.43
729.27
647.79
580.10
523.91
17

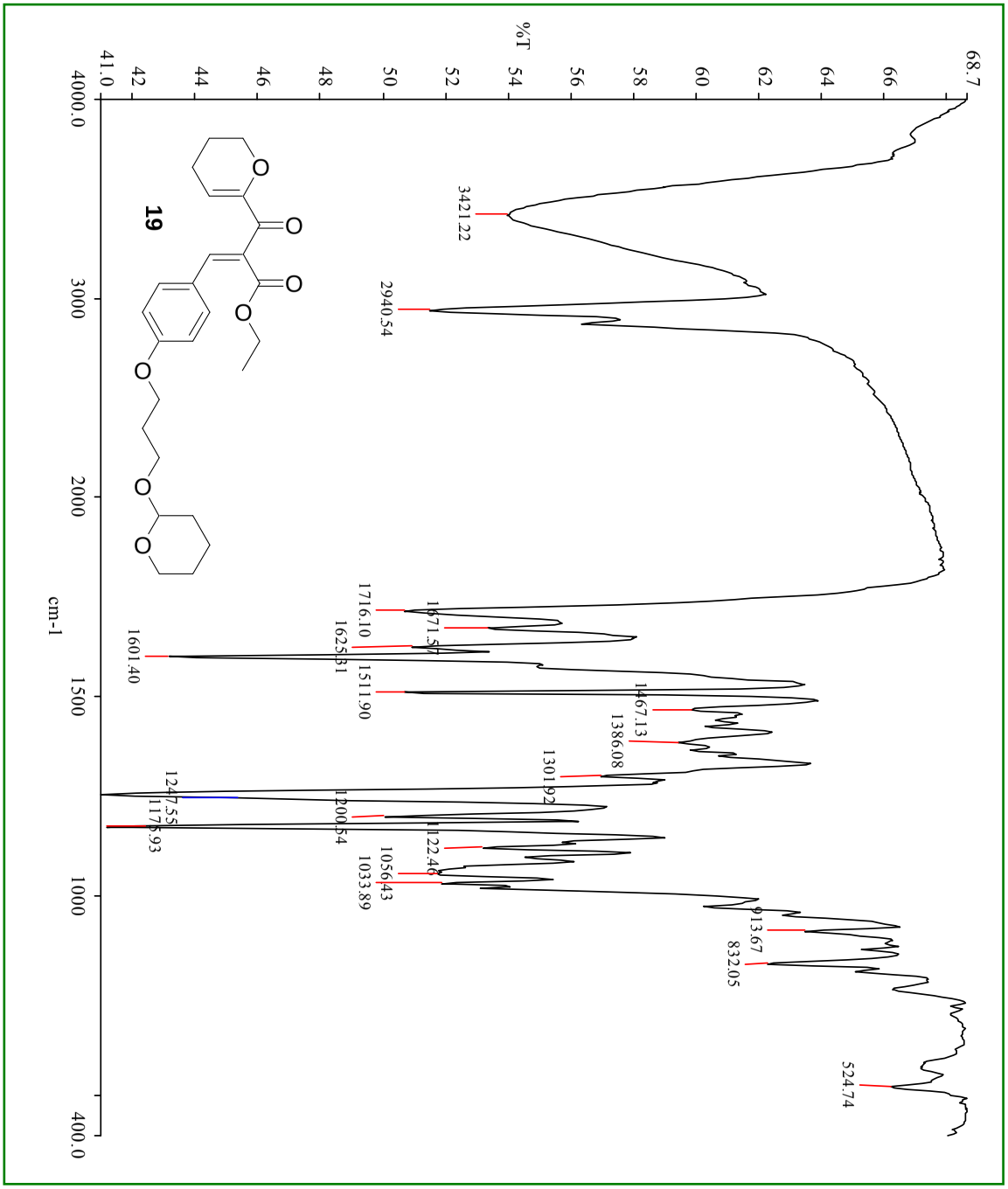

1H
18
7.7404
7.3461
7.3242
6.8433
6.8212
6.0250
6.0144
6.0038
4.2812
4.2636
4.2458
4.2280
4.1213
4.1124
4.1001
4.0872
4.0746
4.0658
4.0510
4.0363
3.5195
3.5032
3.4868
2.2167
2.1716
2.1560
2.1448
2.1293
2.0726
2.0570
2.0415
2.0366
2.0259
2.0104
1.8534
1.8387
1.0000
2.0250
2.0476
0.9902
2.1781
2.1443
2.1356
2.1495
2.0621
2.4640
2.0874
3.2778
ppm
BRUKER
Current Data Parameters
NAME JKIM-382-prefinal azide
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090613
Time_ 11.56
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 3
DS 0
SWH 6393.862 Hz
FIDRES 0.195125 Hz
AQ 2.5625076 sec
RG 161
DW 78.200 usec
DE 6.00 usec
TE 297.1 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.05 usec
PL1 -3.00 dB
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300086 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

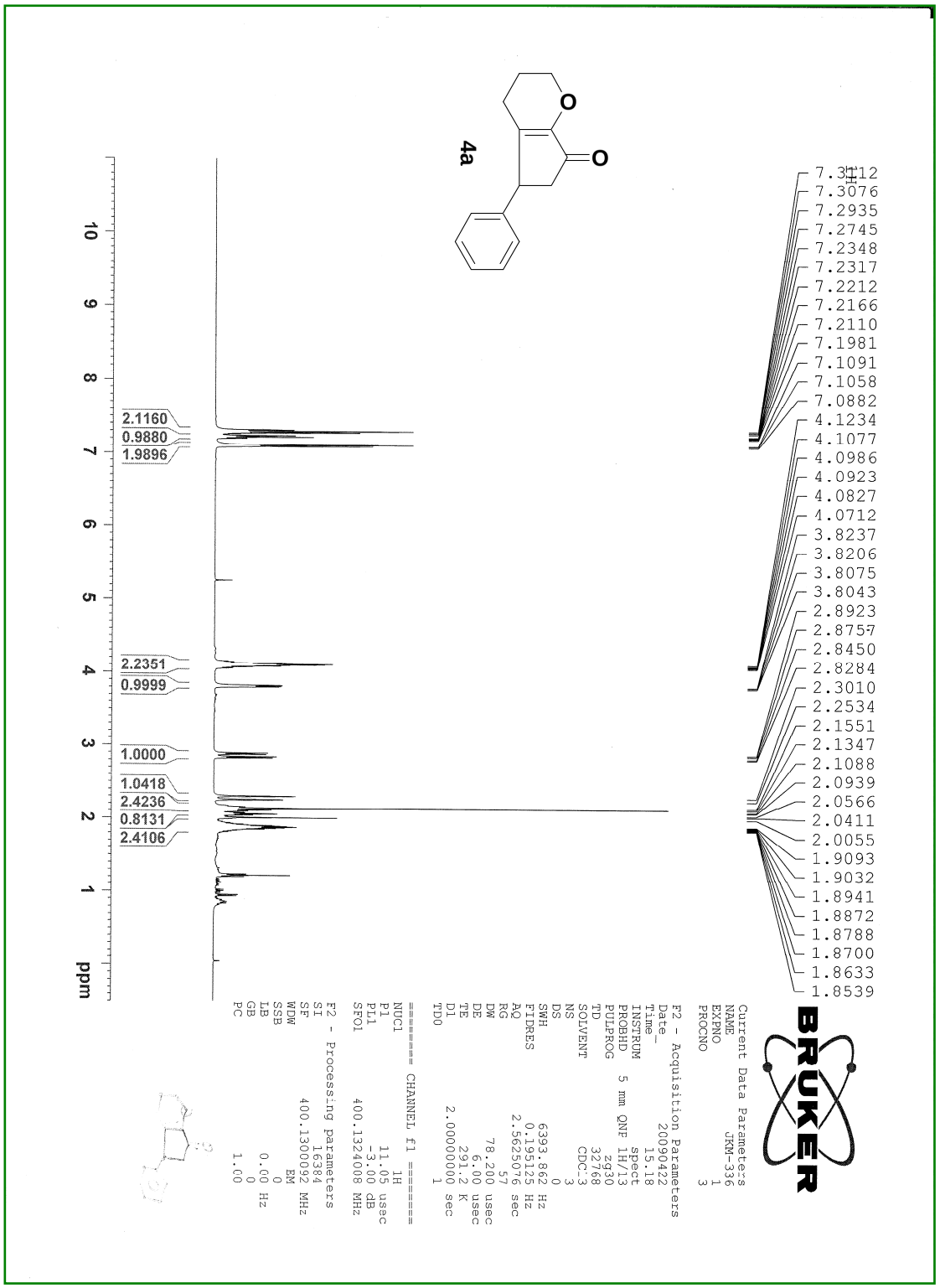

18
13C
191.04
165.20
160.45
151.32
142.58
132.34
128.01
125.86
116.03
114.70
66.54
64.58
61.32
48.09
28.65
21.36
21.05
14.22
ppm
BRUKER

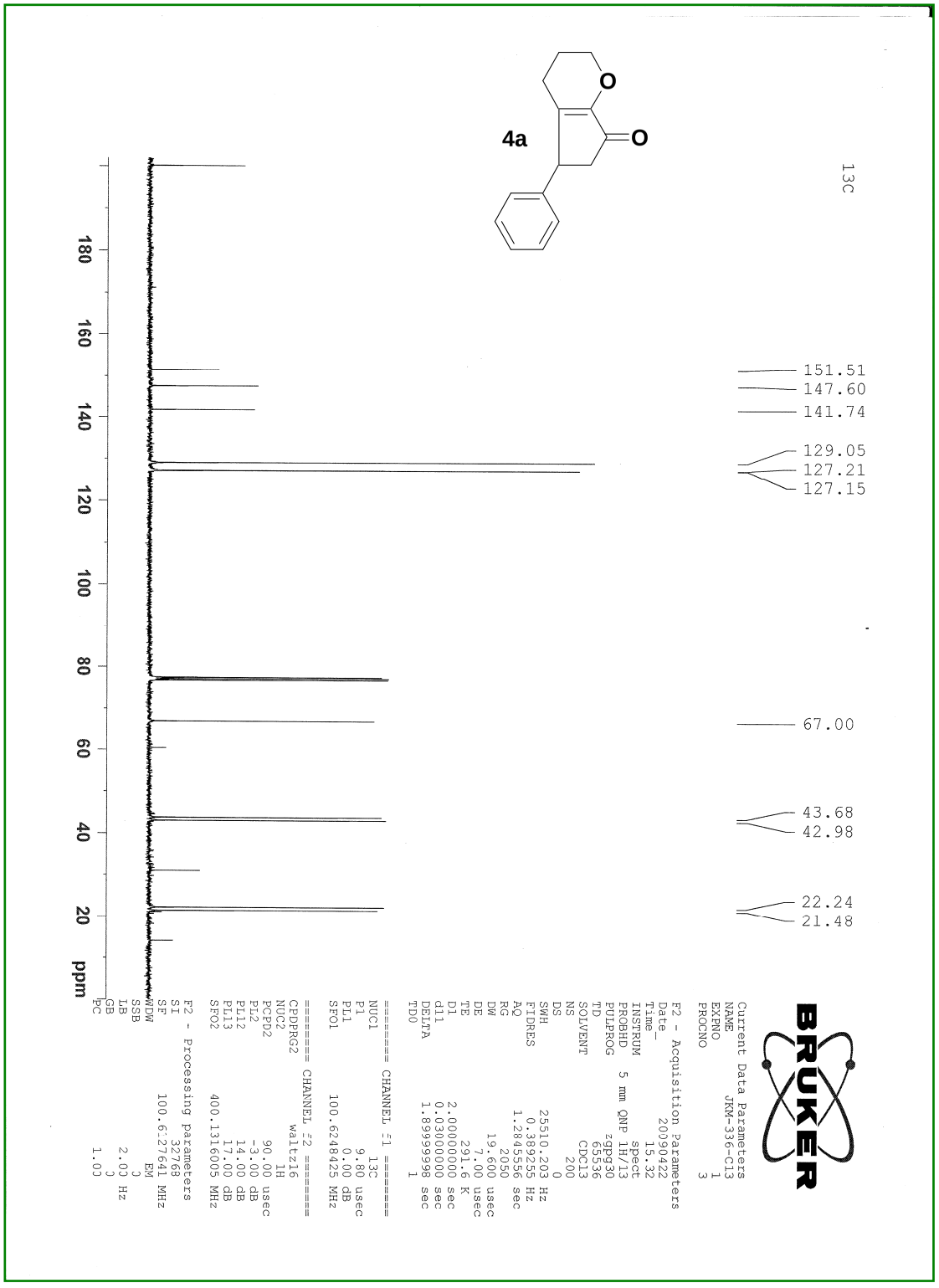


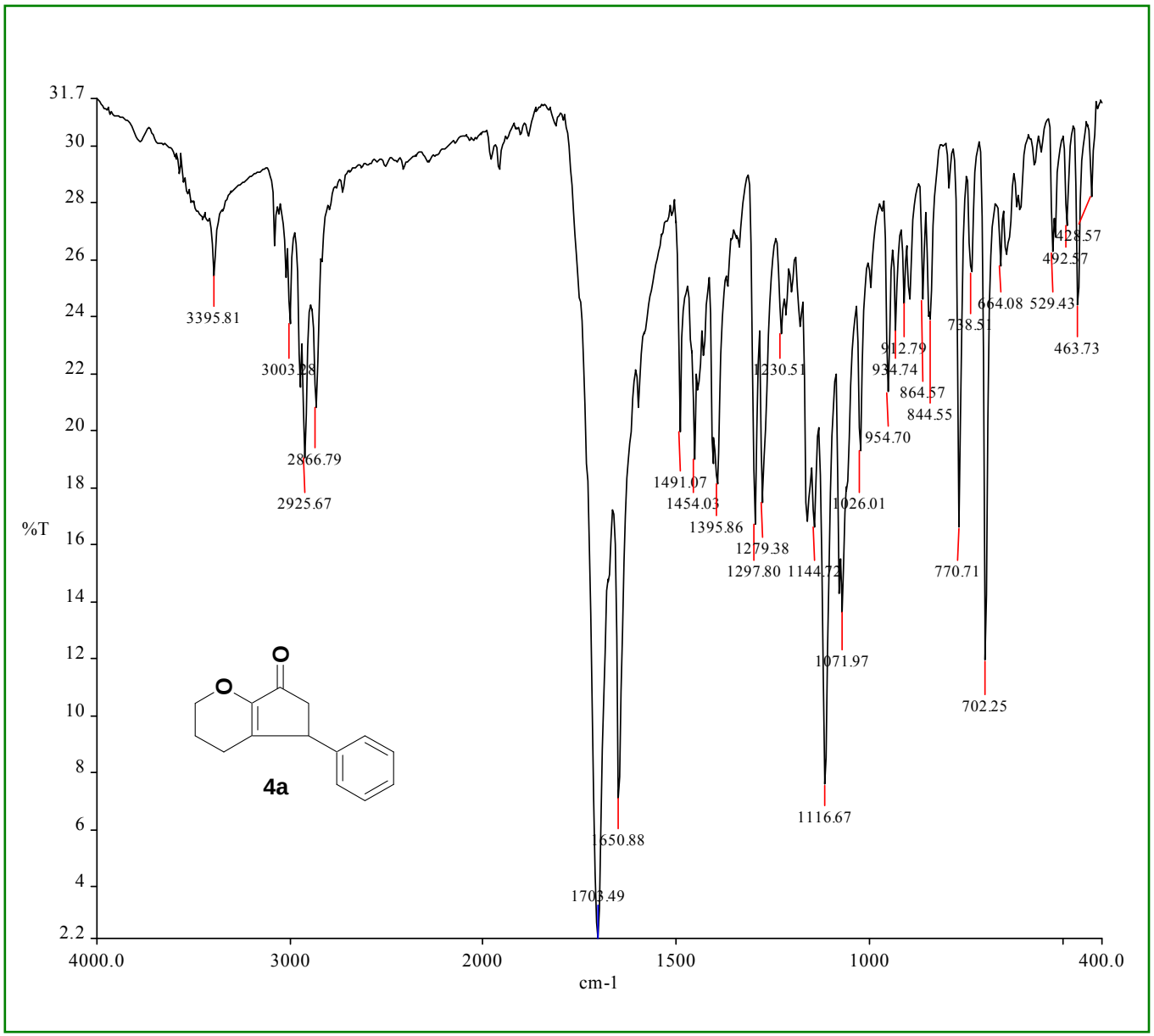

19
0.9351
1.8544
1.9050
0.9202
1.0346
2.0034
4.0000
1.0973
1.0679
1.1115
1.1019
1.9925
2.3468
3.1266
1.2628
4.4694
3.0563
ppm
7.7398
7.3336
7.3116
6.8471
6.8421
6.8297
6.8250
6.0252
6.0146
6.0039
4.5849
4.5782
4.5673
4.2779
4.2601
4.2424
4.2245
4.1136
4.1013
4.0857
4.0689
3.9151
3.8904
3.5651
3.5404
2.1540
2.1430
2.0770
2.0614
2.0459
2.0356
1.8381
1.8264
1.8121
1.5747
1.5389
1.5306
1.5203
1.5034
1.4988
1.2919
1.2740
1.2563
1.2498
Current Data Parameters
NAME JKM-383-prefinal THP
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090617
Time_ 22.48
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 3
DS 0
SWH 6393.862 Hz
FIDRES 0.195125 Hz
AQ 2.5625076 sec
RG 144
DW 78.200 usec
DE 6.00 usec
TE 295.5 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.05 usec
PL1 -3.00 dB
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300089 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

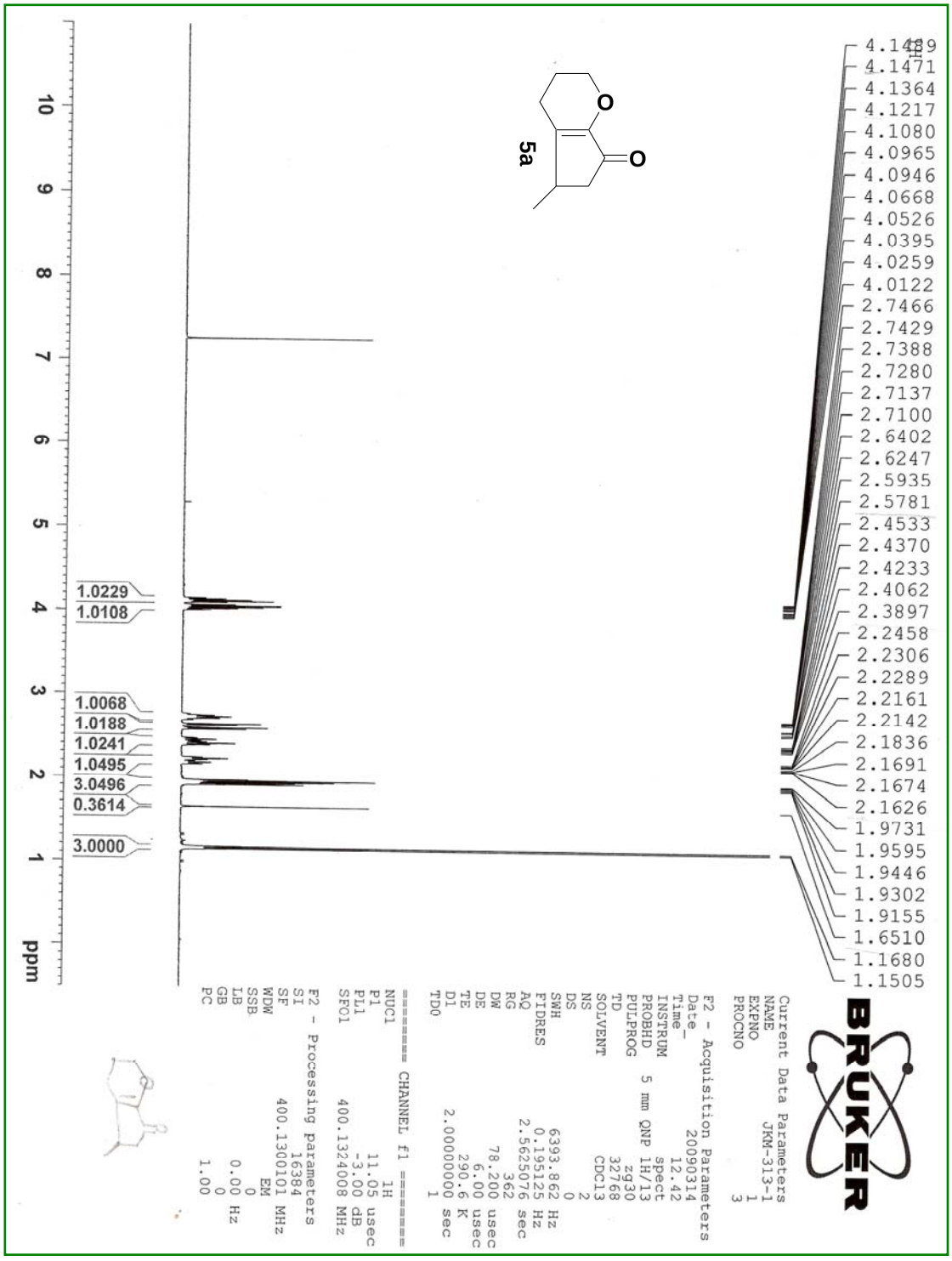

19
13C
191.12
165.26
160.89
151.33
142.77
132.33
127.68
125.49
116.02
114.75
98.99
66.53
65.08
63.81
62.39
61.29
30.67
29.53
25.42
21.36
21.06
19.61
14.23
180
160
140
120
100
80
60
40
20
ppm
BRUKER
Current Data Parameters
NAME JKM-383-pretinal THP-C13
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090617
Time_ 23.02
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 300
DS 0
SWH 25510.203 Hz
FIDRES 0.389255 Hz
AQ 1.2845556 sec
RG 2050
DW 19.600 usec
DE 7.00 usec
TE 296.1 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.80 usec
PL1 0.00 dB
SFO1 100.6248425 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -3.00 dB
PL12 14.00 dB
PL13 17.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00
OTHP

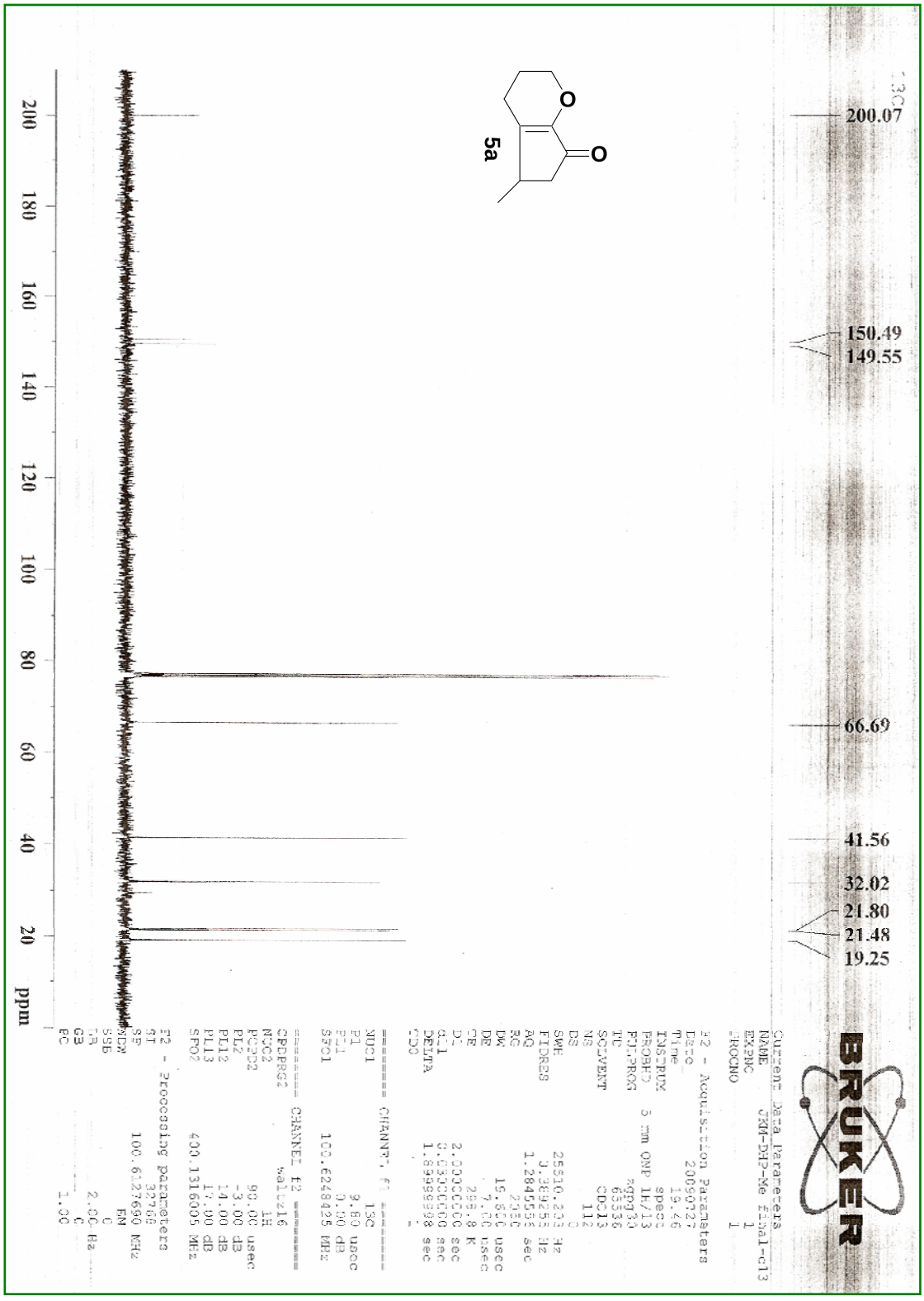

%T
cm-1
19
3421.22
2940.54
1716.10
1671.57
1625.31
1601.40
1511.90
1467.13
1386.08
1301.92
1247.55
1200.54
1176.93
1122.48
1056.43
1033.89
913.67
832.05
524.74

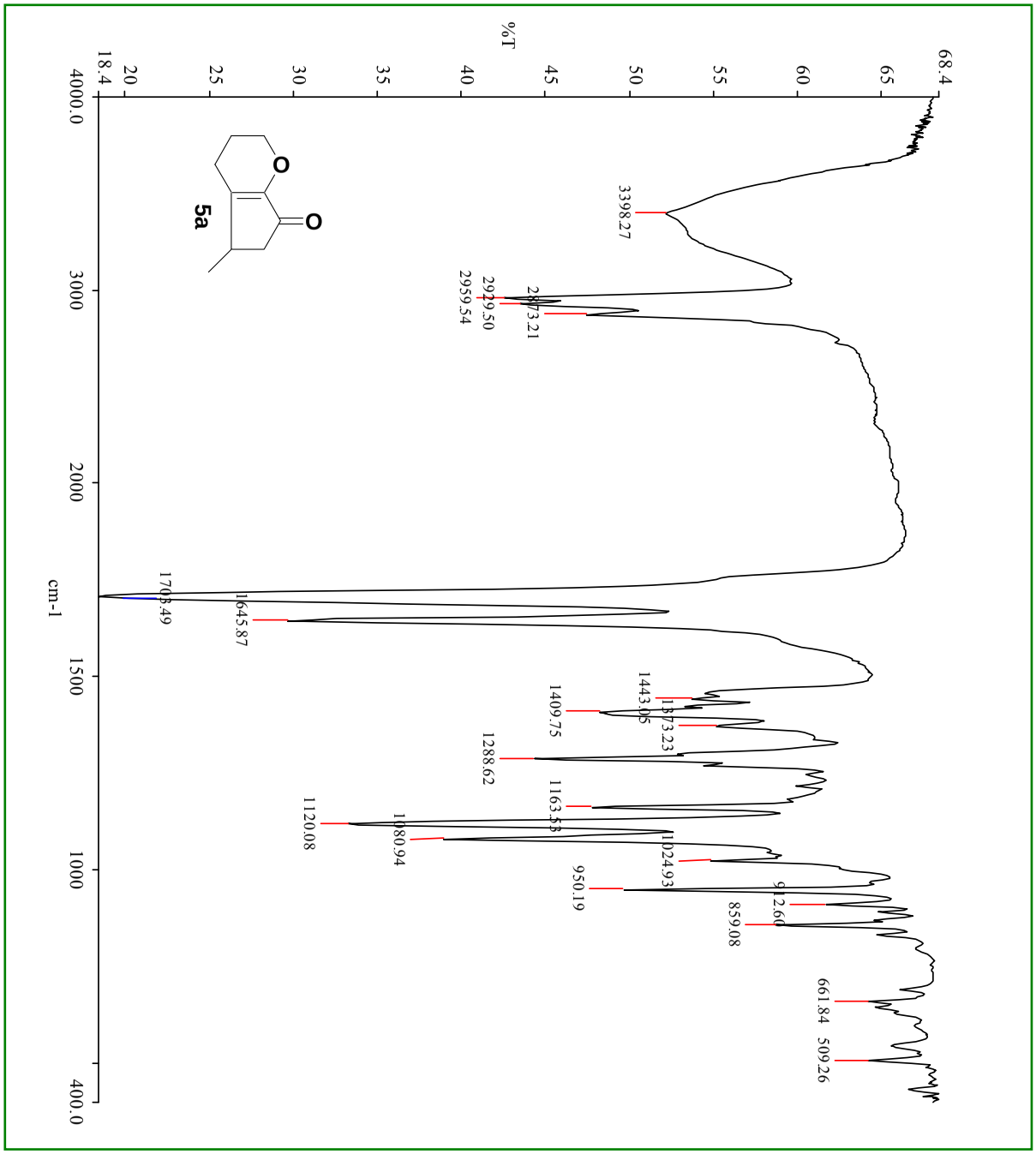

4a
O
O
7.3112
7.3076
7.2935
7.2745
7.2348
7.2317
7.2212
7.2166
7.2110
7.1981
7.1091
7.1058
7.0882
4.1234
4.1077
4.0986
4.0923
4.0827
4.0712
3.8237
3.8206
3.8075
3.8043
2.8923
2.8757
2.8450
2.8284
2.3010
2.2534
2.1551
2.1347
2.1088
2.0939
2.0566
2.0411
2.0055
1.9093
1.9032
1.8941
1.8872
1.8788
1.8700
1.8633
1.8539
2.1160
0.9880
1.9896
2.2351
0.9999
1.0000
1.0418
2.4236
0.8131
2.4106
ppm
BRUKER
Current Data Parameters
NAME JKM-336
EXPNO 1
PROCNO 3
F2 - Acquisition Parameters
Date_ 20090422
Time_ 15.18
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDC:3
NS 3
DS 0
SWH 6393.862 Hz
FIDRES 0.195125 Hz
AQ 2.5625076 sec
RG 57
DW 78.200 usec
DE 6.00 usec
TE 291.2 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.05 usec
PL1 -3.00 dB
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300092 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

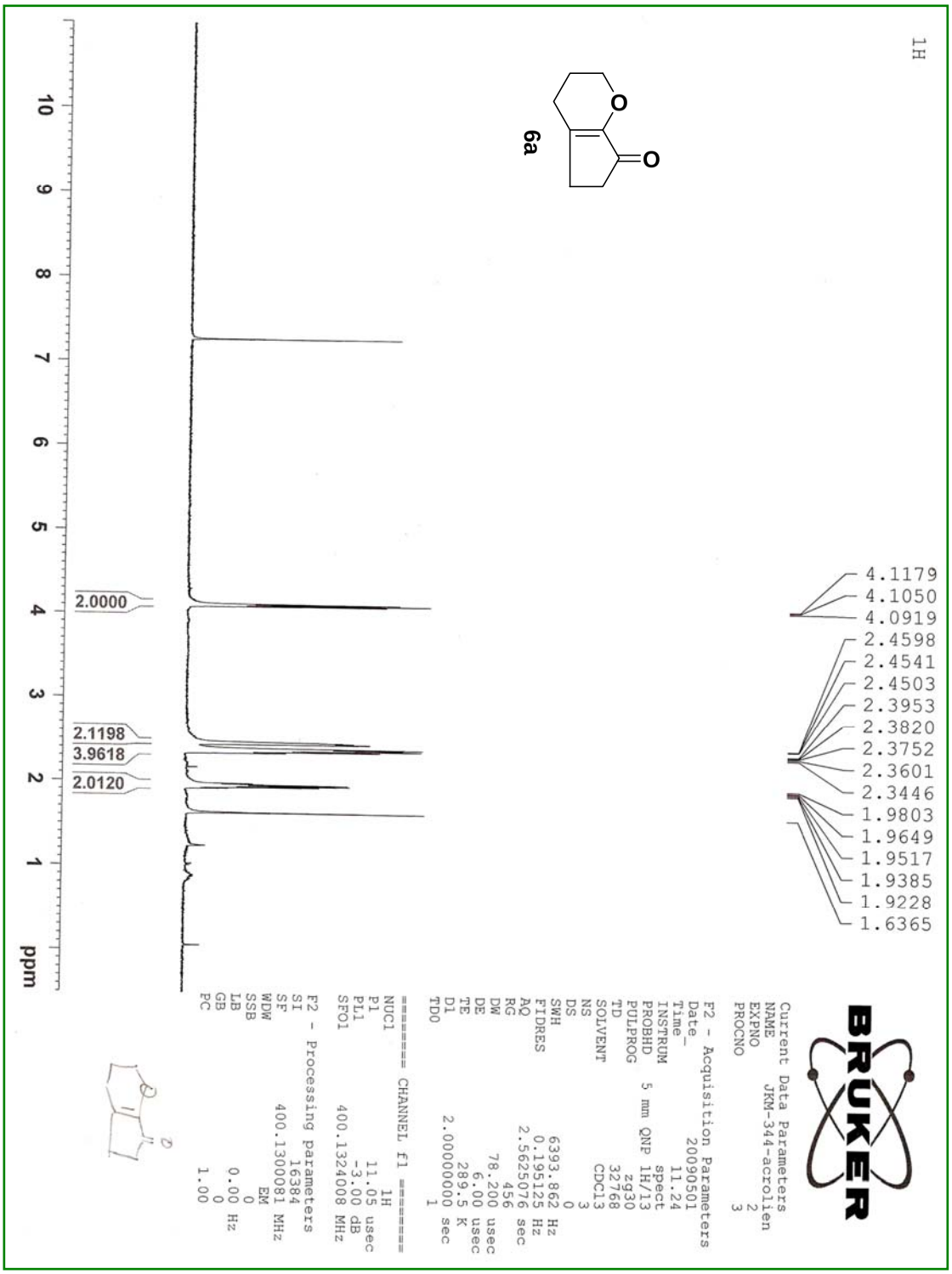

13C
4a
O
O
151.51
147.60
141.74
129.05
127.21
127.15
67.00
43.68
42.98
22.24
21.48
ppm
180
160
140
120
100
80
60
40
20
Current Data Parameters
NAME JRM-336-C13
EXPNO 1
PROCNO 3
F2 - Acquisition Parameters
Date_ 20090422
Time_ 15.32
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 200
DS 0
SWH 25510.203 Hz
FIDRES 0.389255 Hz
AQ 1.2845556 sec
RG 2050
DW 19.600 usec
DE 7.00 usec
TE 291.6 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.80 usec
PL1 0.00 dB
SFO1 100.6240425 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -3.00 dB
PL12 14.00 dB
PL13 17.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127641 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00

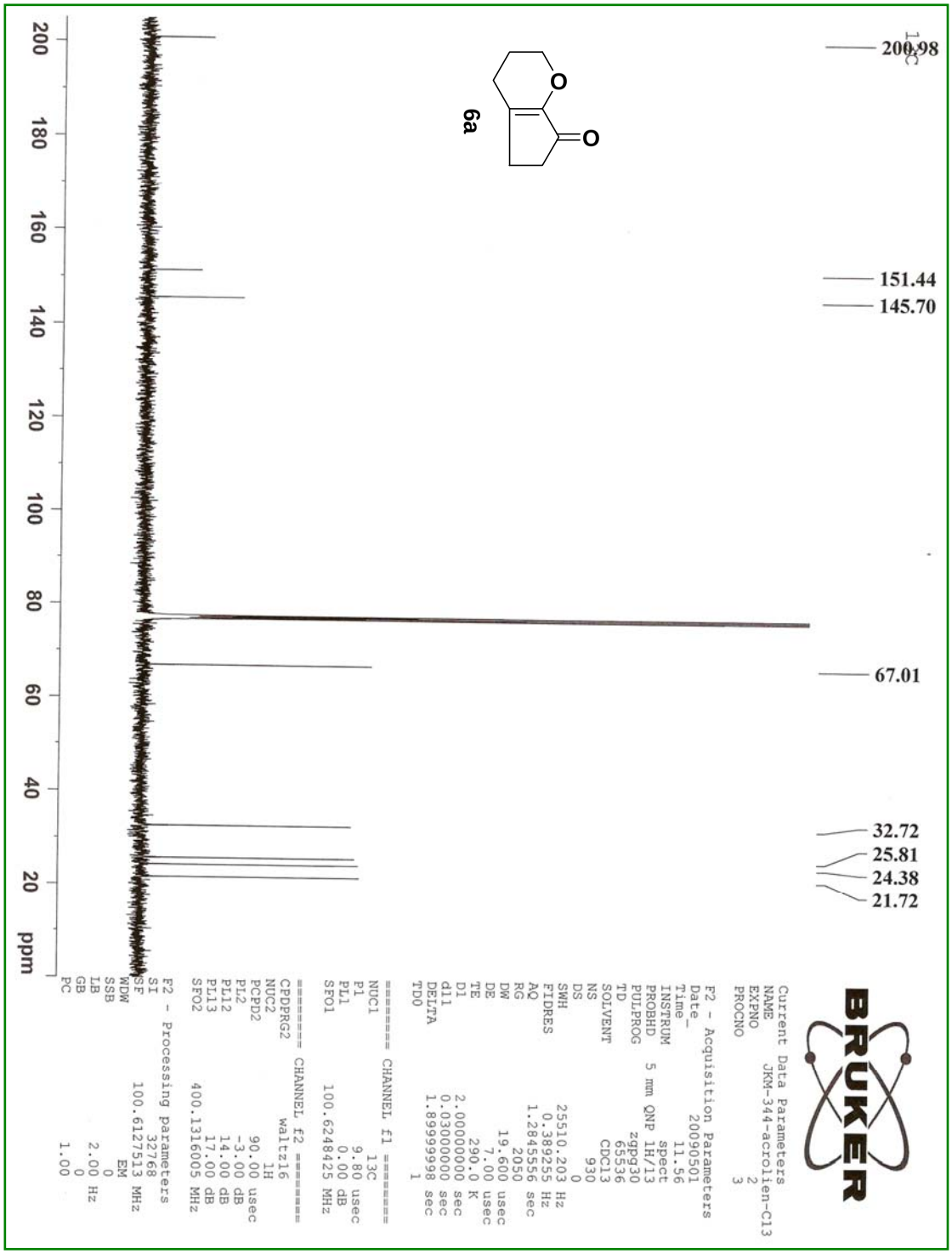


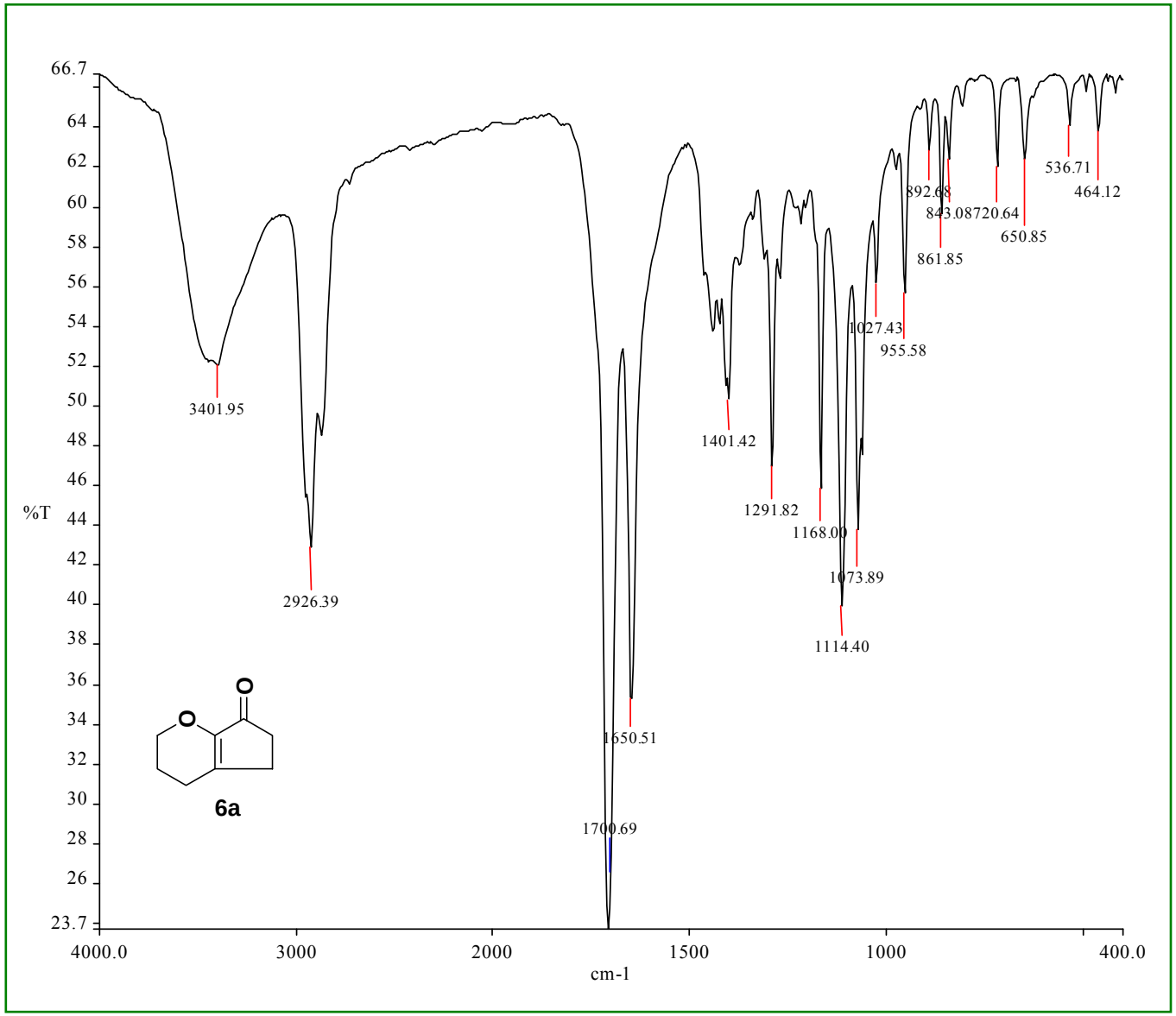

5a
O
O
4.1489
4.1471
4.1364
4.1217
4.1080
4.0965
4.0946
4.0668
4.0526
4.0395
4.0259
4.0122
2.7466
2.7429
2.7388
2.7280
2.7137
2.7100
2.6402
2.6247
2.5935
2.5781
2.4533
2.4370
2.4233
2.4062
2.3897
2.2458
2.2306
2.2289
2.2161
2.2142
2.1836
2.1691
2.1674
2.1626
1.9731
1.9595
1.9446
1.9302
1.9155
1.6510
1.1680
1.1505
1.0229
1.0108
1.0068
1.0188
1.0241
1.0495
3.0496
0.3614
3.0000
10
9
8
7
6
5
4
3
2
1
ppm
Current Data Parameters
NAME JKM-313-1
EXPNO 1
PROCNO 3
F2 - Acquisition Parameters
Date_ 20090314
Time_ 12.42
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 2
DS 0
SWH 6393.862 Hz
FIDRES 0.195125 Hz
AQ 2.5625076 sec
RG 362
DW 78.200 usec
DE 6.00 usec
TE 290.6 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.05 usec
PL1 -3.00 dB
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300101 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
BRUKER

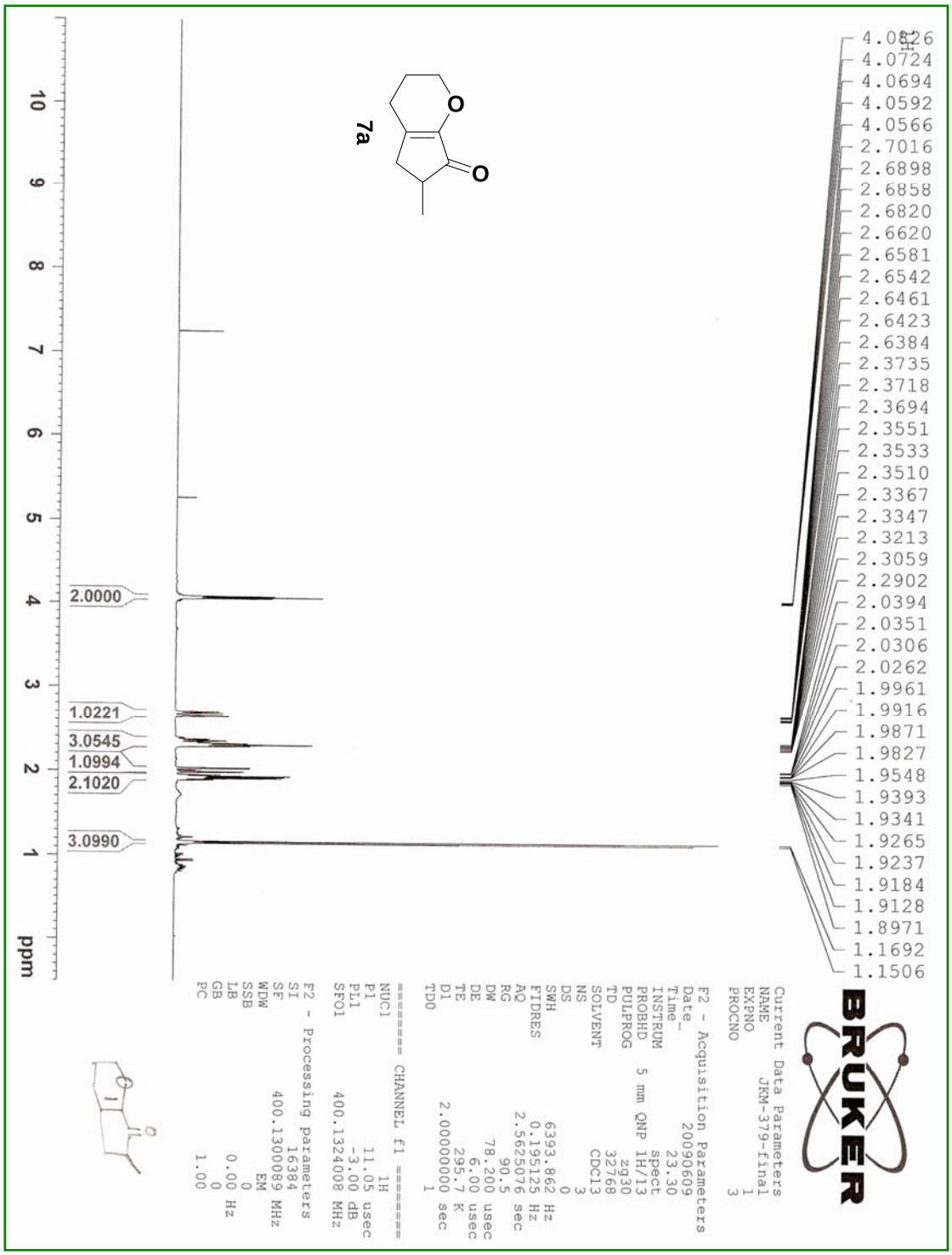

5a
200.07
150.49
149.55
66.69
41.56
32.02
21.80
21.48
19.25
ppm
BRUKER

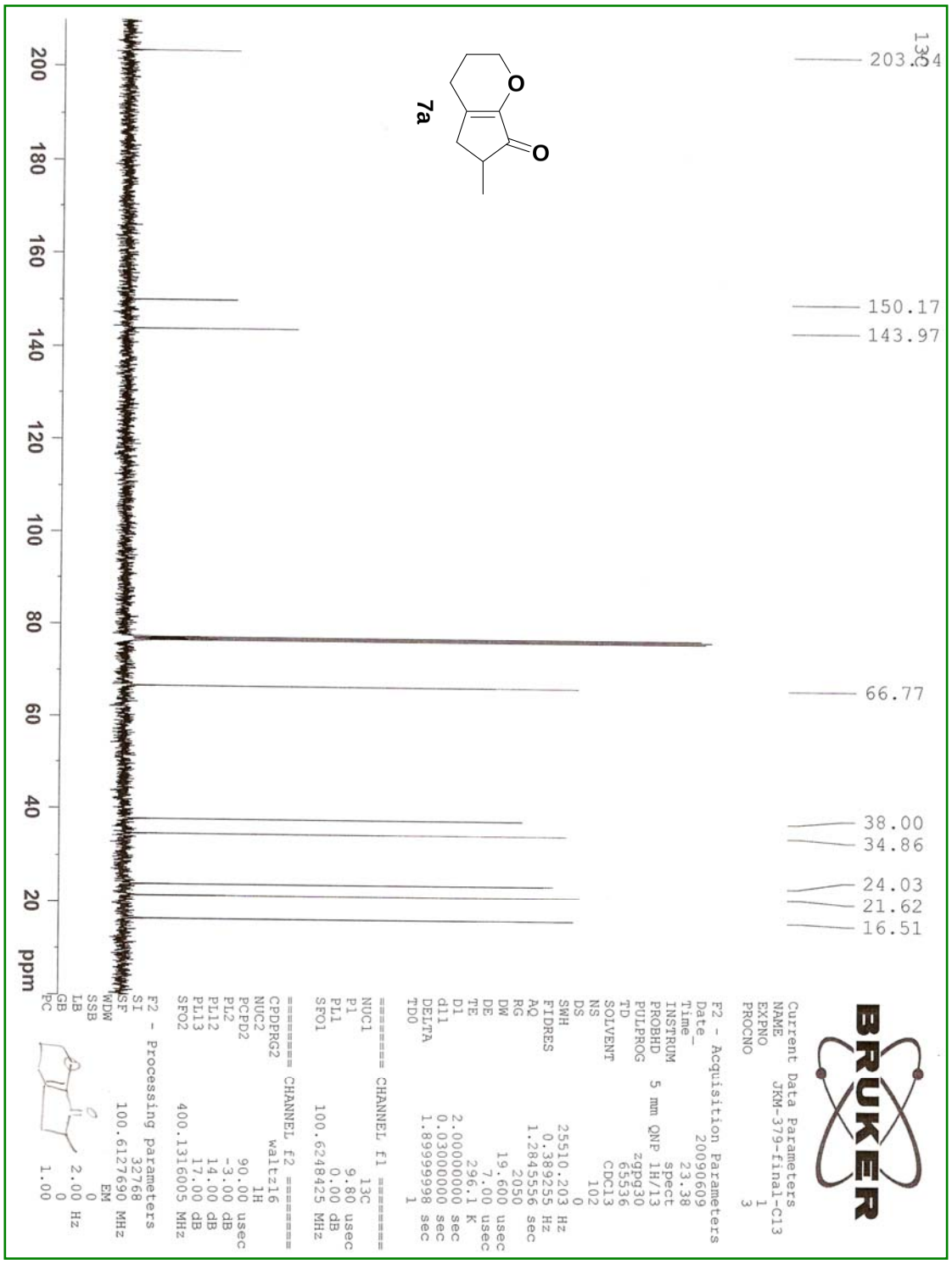

%T
cm-1
68.4
65
60
55
50
45
40
35
30
25
20
18.4
4000.0
3000
2000
1500
1000
400.0
3398.27
2959.54
2929.50
2873.21
1708.49
1645.87
1443.05
1373.23
1409.75
1288.62
1163.53
1120.08
1080.94
1024.93
950.19
912.60
859.08
661.84
509.26
O
O
5a

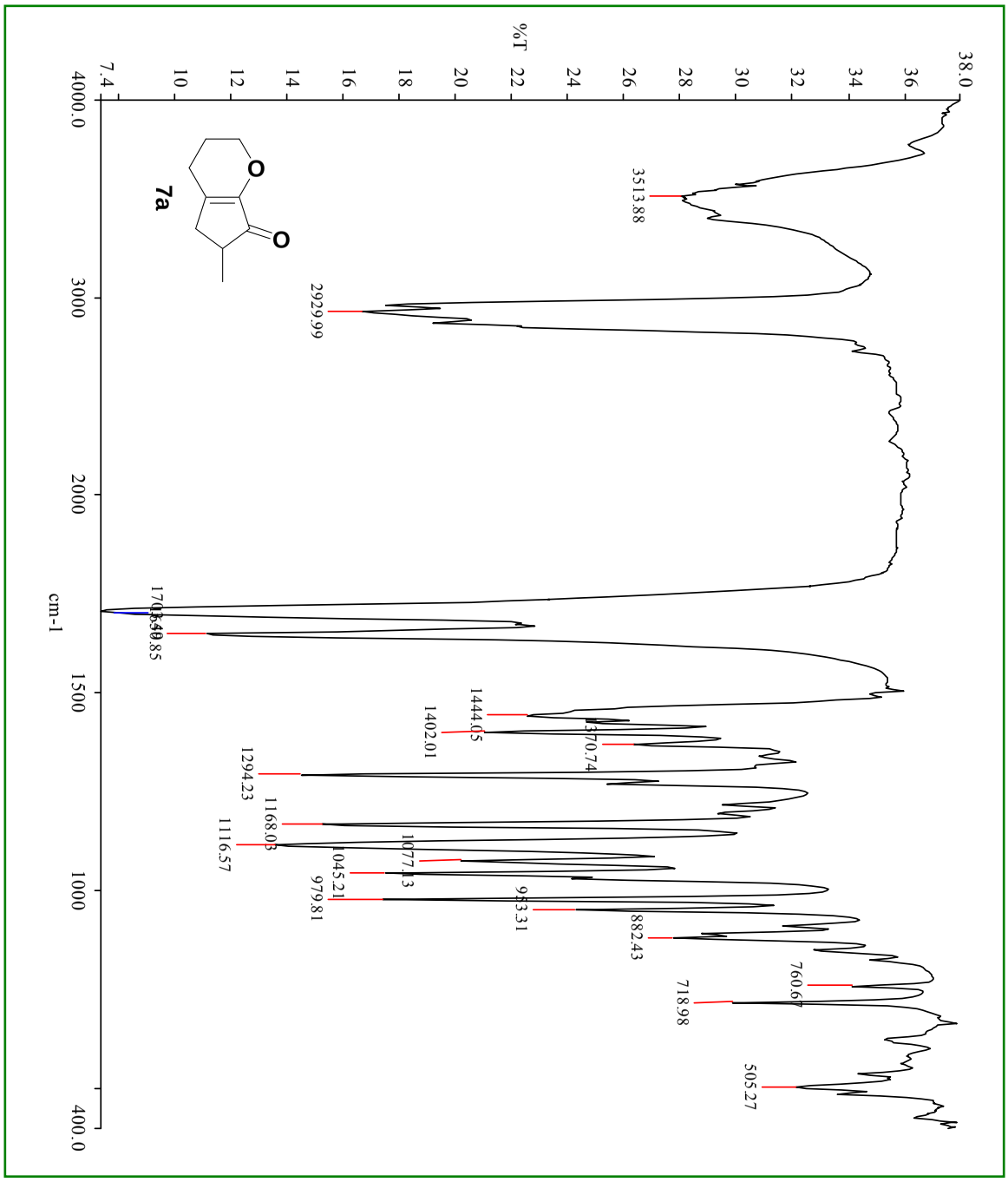

1H
6a
O
O
4.1179
4.1050
4.0919
2.4598
2.4541
2.4503
2.3953
2.3820
2.3752
2.3601
2.3446
1.9803
1.9649
1.9517
1.9385
1.9228
1.6365
2.0000
2.1198
3.9618
2.0120
10 9 8 7 6 5 4 3 2 1 ppm
BRUKER
Current Data Parameters
NAME JKM-344-acrolien
EXPNO 2
PROCNO 3
F2 - Acquisition Parameters
Date_ 20090501
Time_ 11.24
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 3
DS 0
SWH 6393.862 Hz
FIDRES 0.195125 Hz
AQ 2.5625076 sec
RG 456
DW 78.200 usec
DE 6.00 usec
TE 289.5 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.05 usec
PL1 -3.00 dB
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300081 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

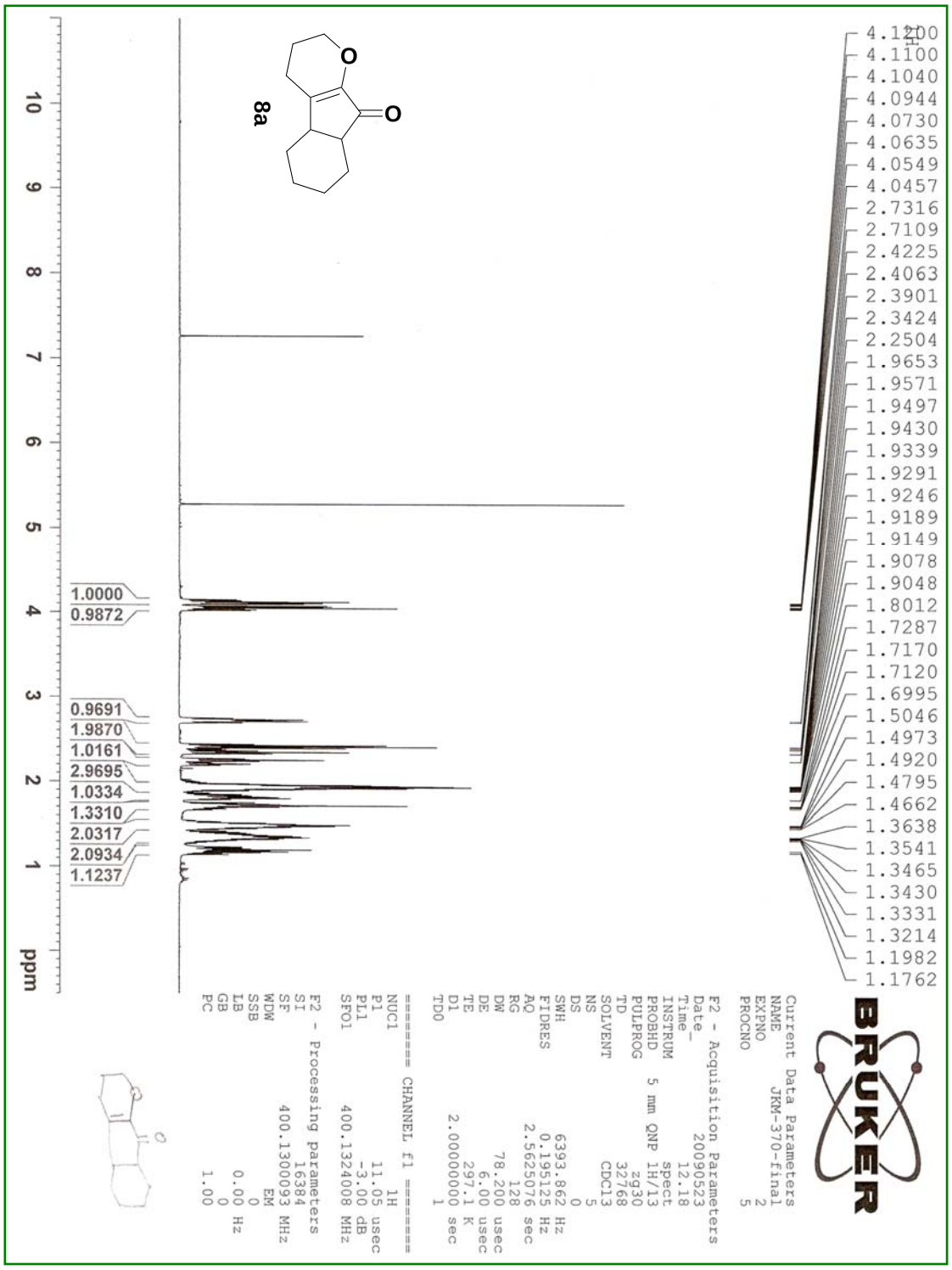

6a
O
O
200.98
151.44
145.70
67.01
32.72
25.81
24.38
21.72
200
180
160
140
120
100
80
60
40
20
ppm
BRUKER
Current Data Parameters
NAME JKM-344-acrolien-C13
EXPNO 2
PROCNO 3
F2 - Acquisition Parameters
Date_ 20090501
Time_ 11.56
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 930
DS 0
SWH 25510.203 Hz
FIDRES 0.389255 Hz
AQ 1.2845556 sec
RG 2050
DW 19.600 usec
DE 7.00 usec
TE 290.0 K
D1 2.00000000 sec
D11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.80 usec
PL1 0.00 dB
SFO1 100.6248425 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -3.00 dB
PL12 14.00 dB
PL13 17.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127513 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00

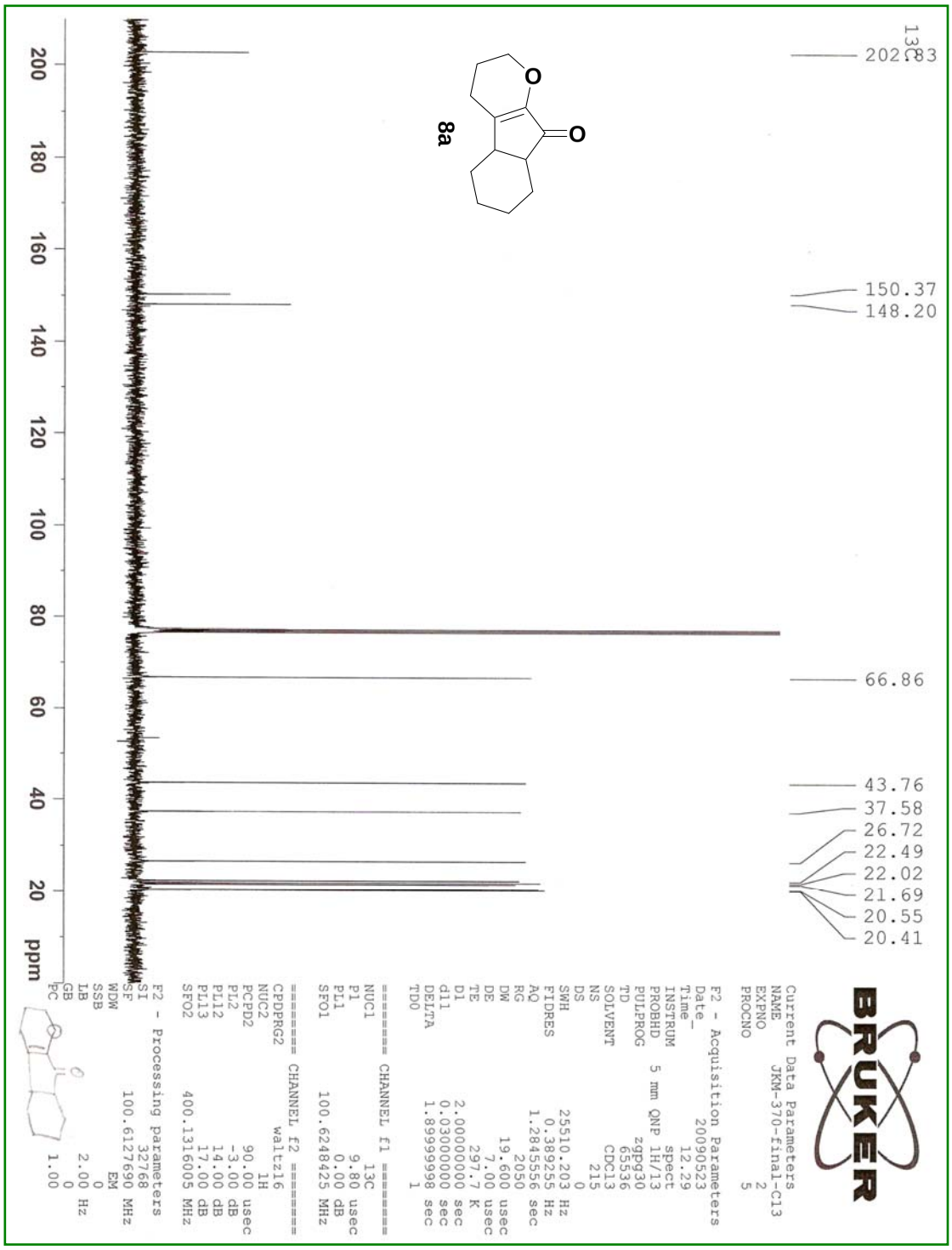


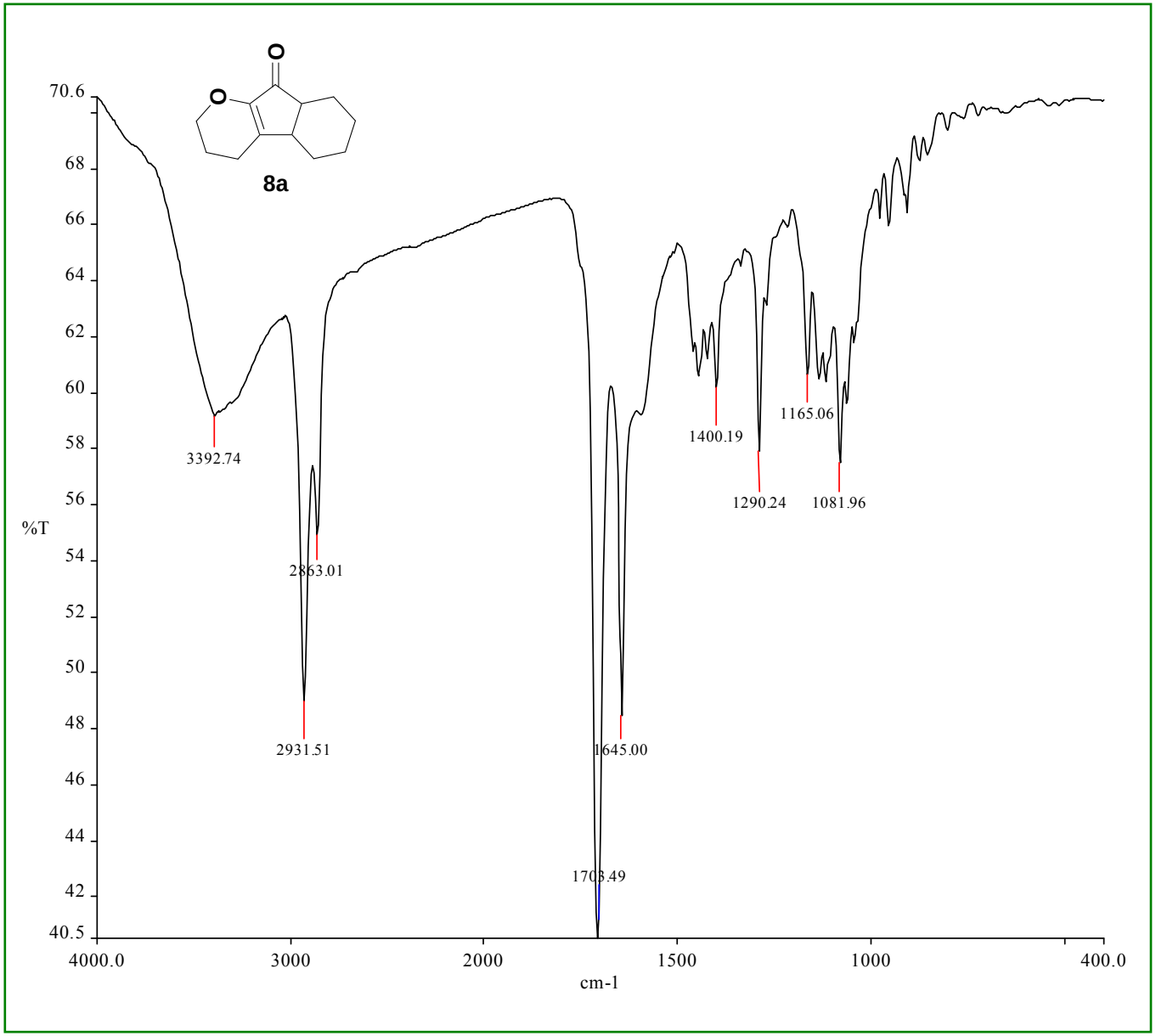

7a
O
O
4.0826
4.0724
4.0694
4.0592
4.0566
2.7016
2.6898
2.6858
2.6820
2.6620
2.6581
2.6542
2.6461
2.6423
2.6384
2.3735
2.3718
2.3694
2.3551
2.3533
2.3510
2.3367
2.3347
2.3213
2.3059
2.2902
2.0394
2.0351
2.0306
2.0262
1.9961
1.9916
1.9871
1.9827
1.9548
1.9393
1.9341
1.9265
1.9237
1.9184
1.9128
1.8971
1.1692
1.1506
2.0000
1.0221
3.0545
1.0994
2.1020
3.0990
ppm
BRUKER
Current Data Parameters
NAME JRM-379-final
EXPNO 1
PROCNO 3
F2 - Acquisition Parameters
Date_ 20090609
Time_ 23.30
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 3
DS 0
SWH 6393.862 Hz
FIDRES 0.195125 Hz
AQ 2.5625076 sec
RG 90.5
DW 78.200 usec
DE 6.00 usec
TE 295.7 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.05 usec
PL1 -3.00 dB
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300089 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

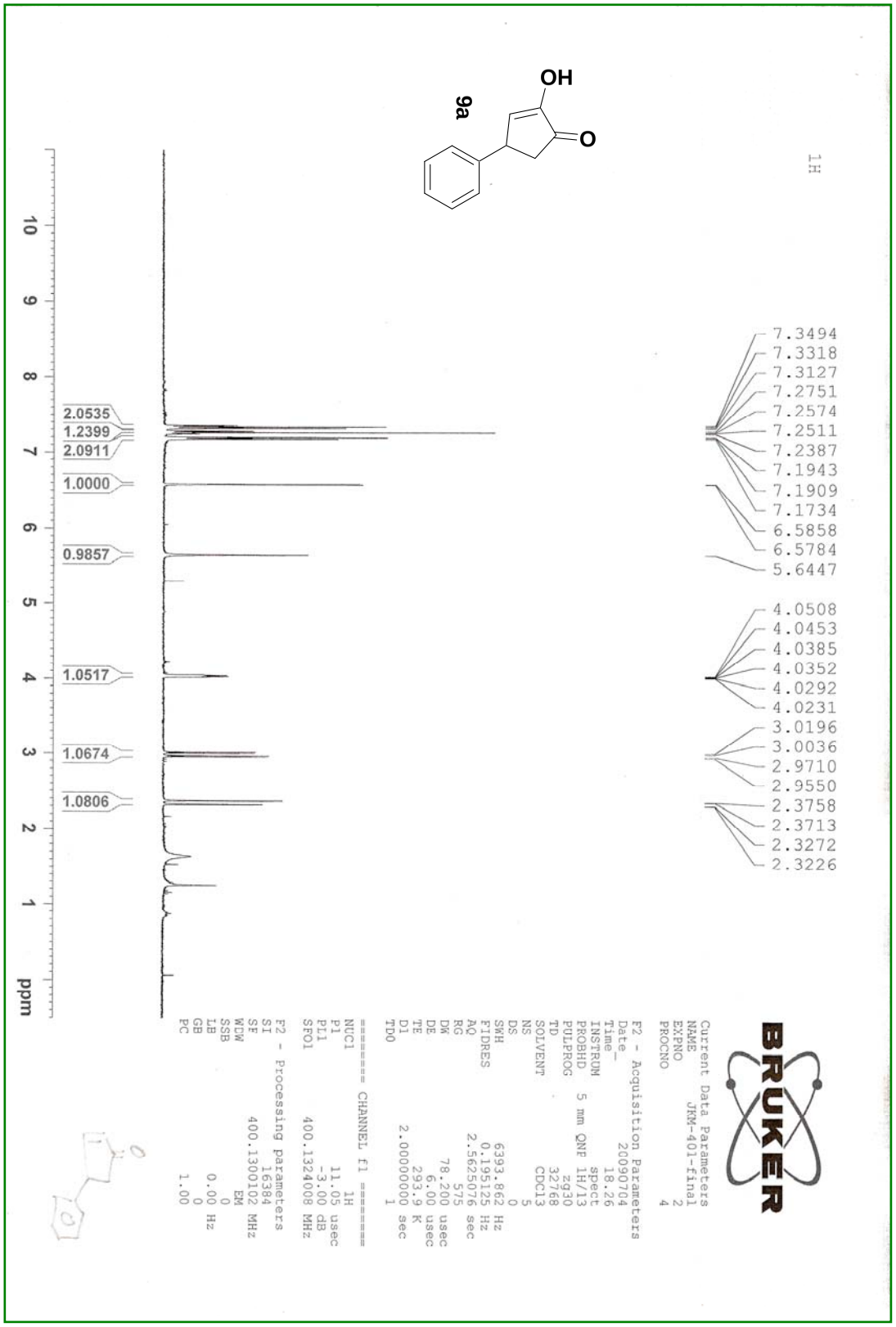

7a
203.34
150.17
143.97
66.77
38.00
34.86
24.03
21.62
16.51
ppm
Current Data Parameters
NAME JKM-379-final-C13
EXPNO 1
PROCNO 3
F2 - Acquisition Parameters
Date_ 20090609
Time_ 23.38
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 102
DS 0
SWH 25510.203 Hz
FIDRES 0.389255 Hz
AQ 1.2845556 sec
RG 2050
DW 19.600 usec
DE 7.00 usec
TE 296.1 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.80 usec
PL1 0.00 dB
SFO1 100.6248425 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -3.00 dB
PL12 14.00 dB
PL13 17.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00

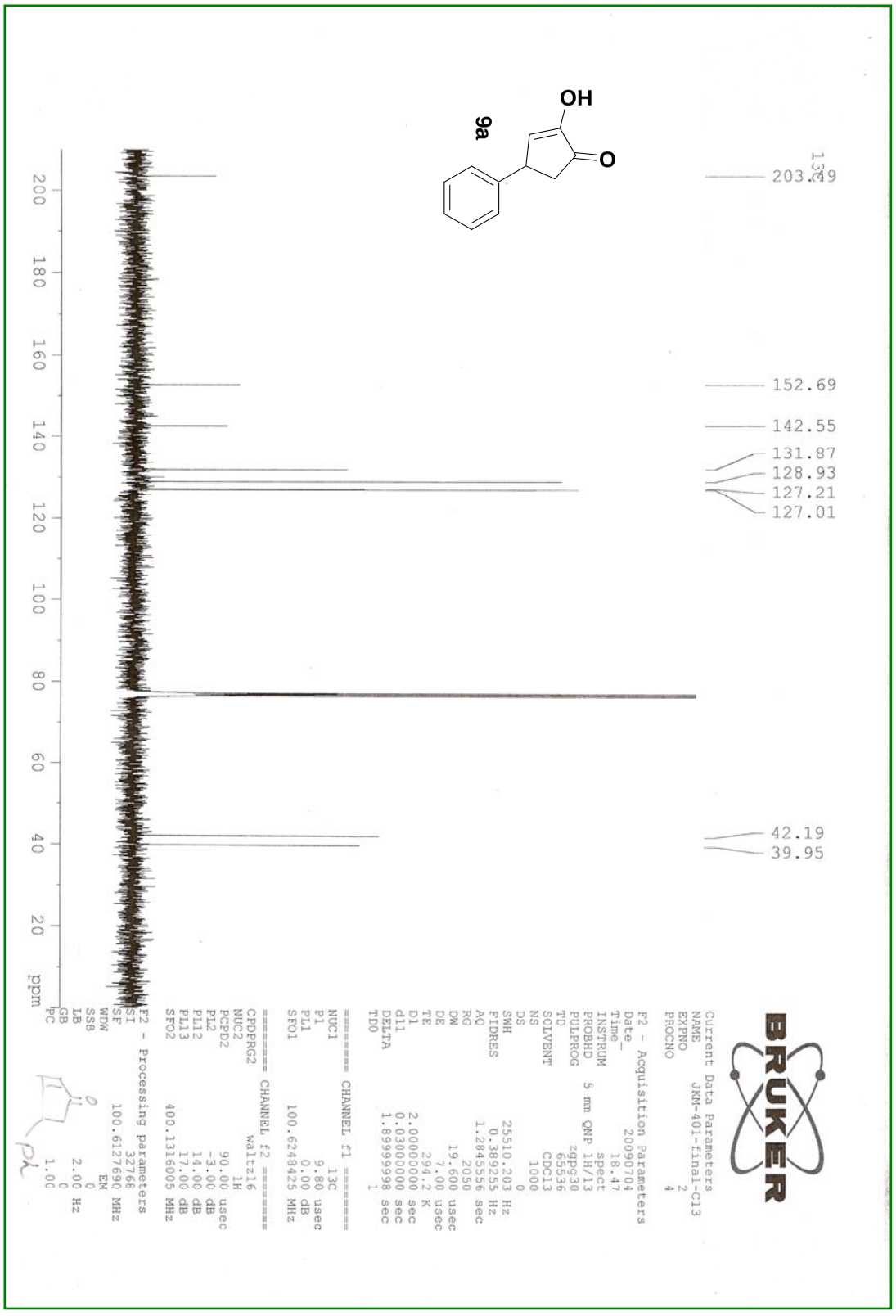

7a
O
O
%T
cm-1
38.0
36
34
32
30
28
26
24
22
20
18
16
14
12
10
7.4
4000.0
3000
2000
1500
1000
400.0
3513.88
2929.99
1444.05
1402.01
1370.74
1294.23
1168.03
1116.57
1077.13
1045.21
979.81
933.31
882.43
760.67
718.98
505.27

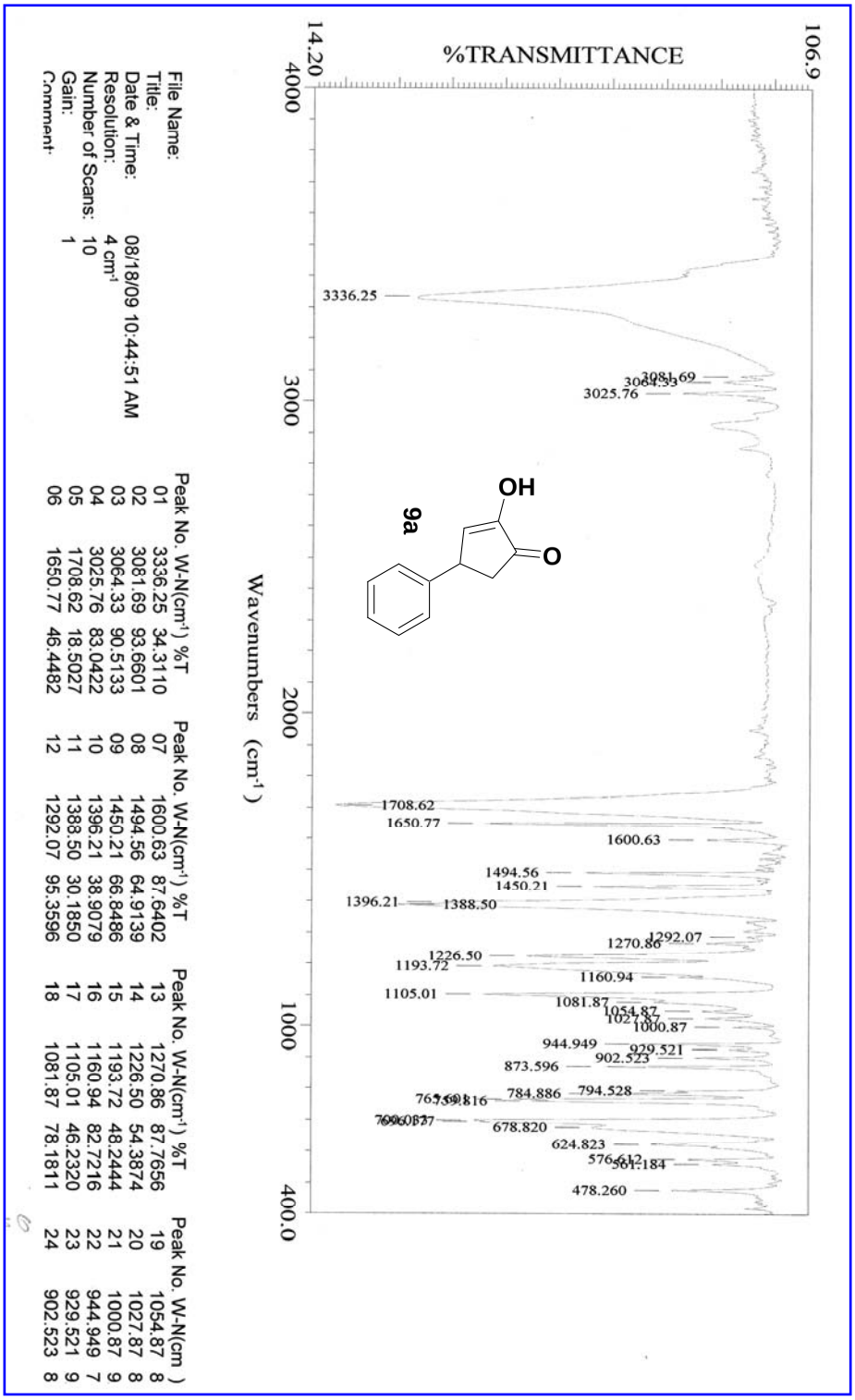

8a
O
O
1.0000
0.9872
0.9691
1.9870
1.0161
2.9695
1.0334
1.3310
2.0317
2.0934
1.1237
ppm
4.1200
4.1100
4.1040
4.0944
4.0730
4.0635
4.0549
4.0457
2.7316
2.7109
2.4225
2.4063
2.3901
2.3424
2.2504
1.9653
1.9571
1.9497
1.9430
1.9339
1.9291
1.9246
1.9189
1.9149
1.9078
1.9048
1.8012
1.7287
1.7170
1.7120
1.6995
1.5046
1.4973
1.4920
1.4795
1.4662
1.3638
1.3541
1.3465
1.3430
1.3331
1.3214
1.1982
1.1762
Current Data Parameters
NAME JKM-370-final
EXPNO 2
PROCNO 5
F2 - Acquisition Parameters
Date_ 20090523
Time_ 12.18
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 5
DS 0
SWH 6393.862 Hz
FIDRES 0.195125 Hz
AQ 2.5625076 sec
RG 128
DW 78.200 usec
DE 6.00 usec
TE 297.1 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.05 usec
PL1 -3.00 dB
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300093 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
BRUKER

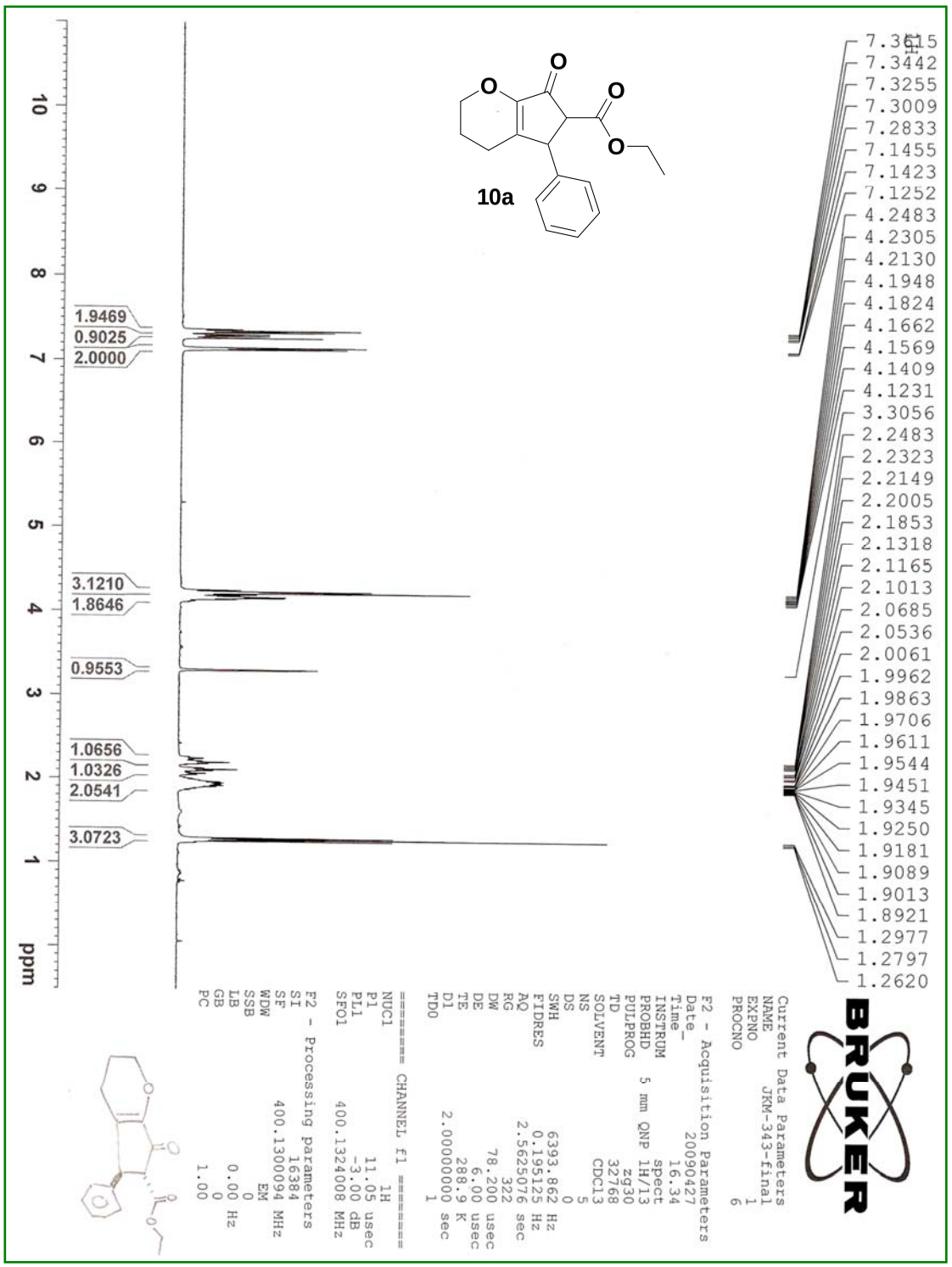

8a
202.83
150.37
148.20
66.86
43.76
37.58
26.72
22.49
22.02
21.69
20.55
20.41
200
180
160
140
120
100
80
60
40
20
ppm
BRUKER

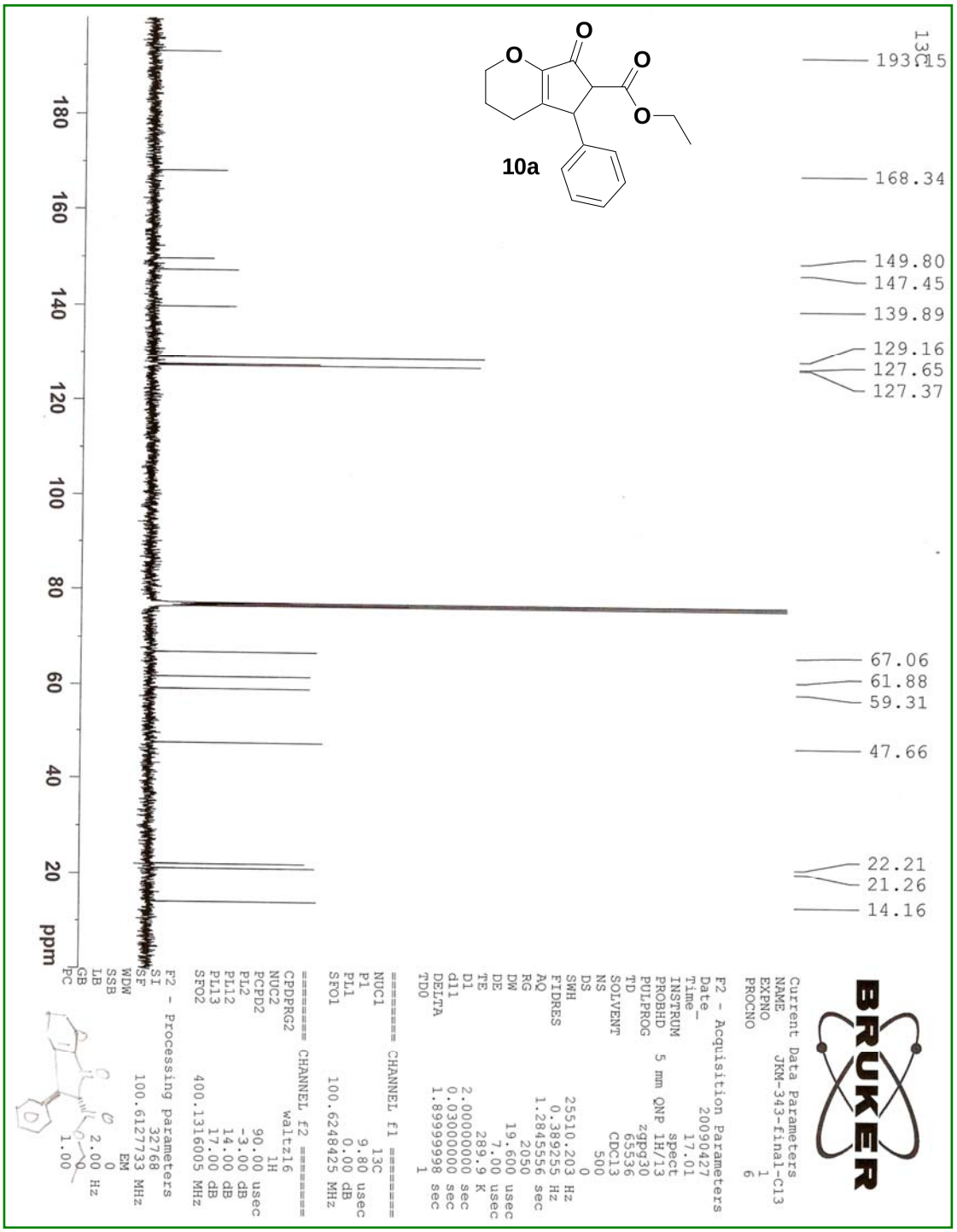


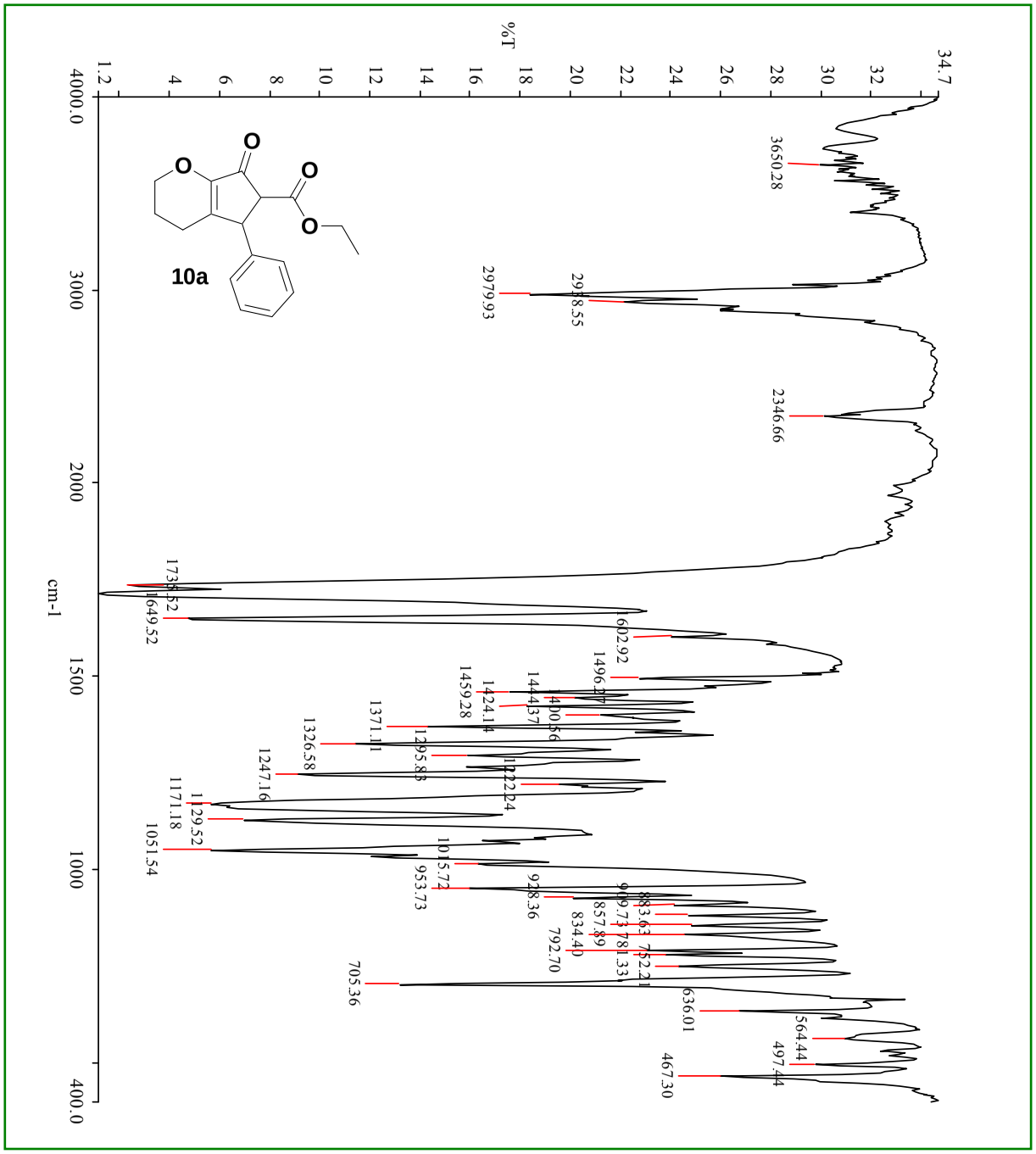

OH
9a
O
1H
7.3494
7.3318
7.3127
7.2751
7.2574
7.2511
7.2387
7.1943
7.1909
7.1734
6.5858
6.5784
5.6447
4.0508
4.0453
4.0385
4.0352
4.0292
4.0231
3.0196
3.0036
2.9710
2.9550
2.3758
2.3713
2.3272
2.3226
2.0535
1.2399
2.0911
1.0000
0.9857
1.0517
1.0674
1.0806
ppm
BRUKER

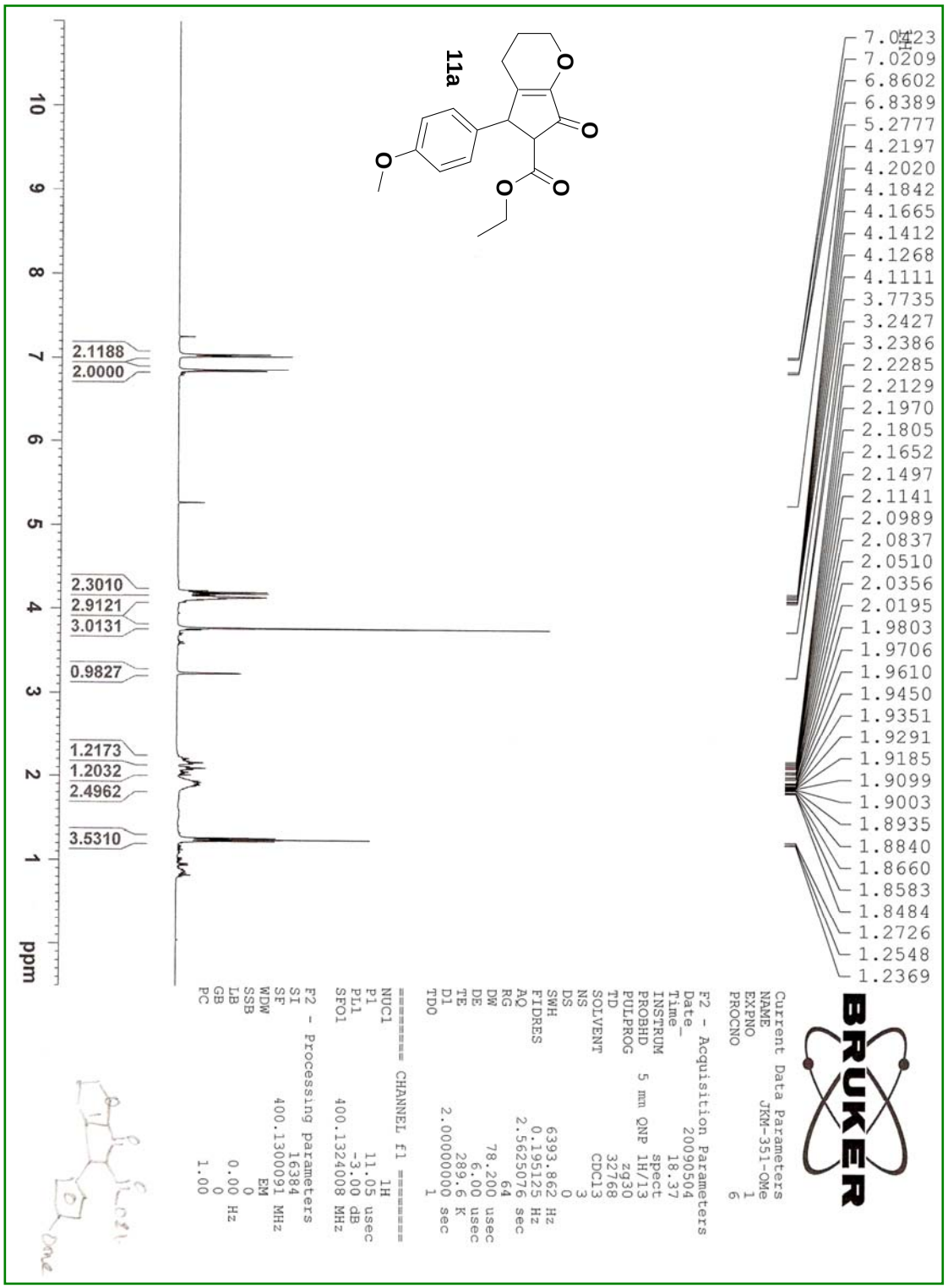

OH
O
9a
203.49
152.69
142.55
131.87
128.93
127.21
127.01
42.19
39.95
ppm
BRUKER
Current Data Parameters
NAME JRM-401-final-C13
EXPNO 2
PROCNO 4
F2 - Acquisition Parameters
Date_ 20090704
Time 18.47
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1000
DS 0
SWH 25510.203 Hz
FIDRES 0.389255 Hz
AQ 1.2843556 sec
RG 2050
DW 19.600 usec
DE 7.00 usec
TE 294.2 K
D1 2.00000000 sec
D11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.80 usec
PL1 0.00 dB
SFO1 100.6248425 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -3.00 dB
PL12 14.00 dB
PL13 17.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00

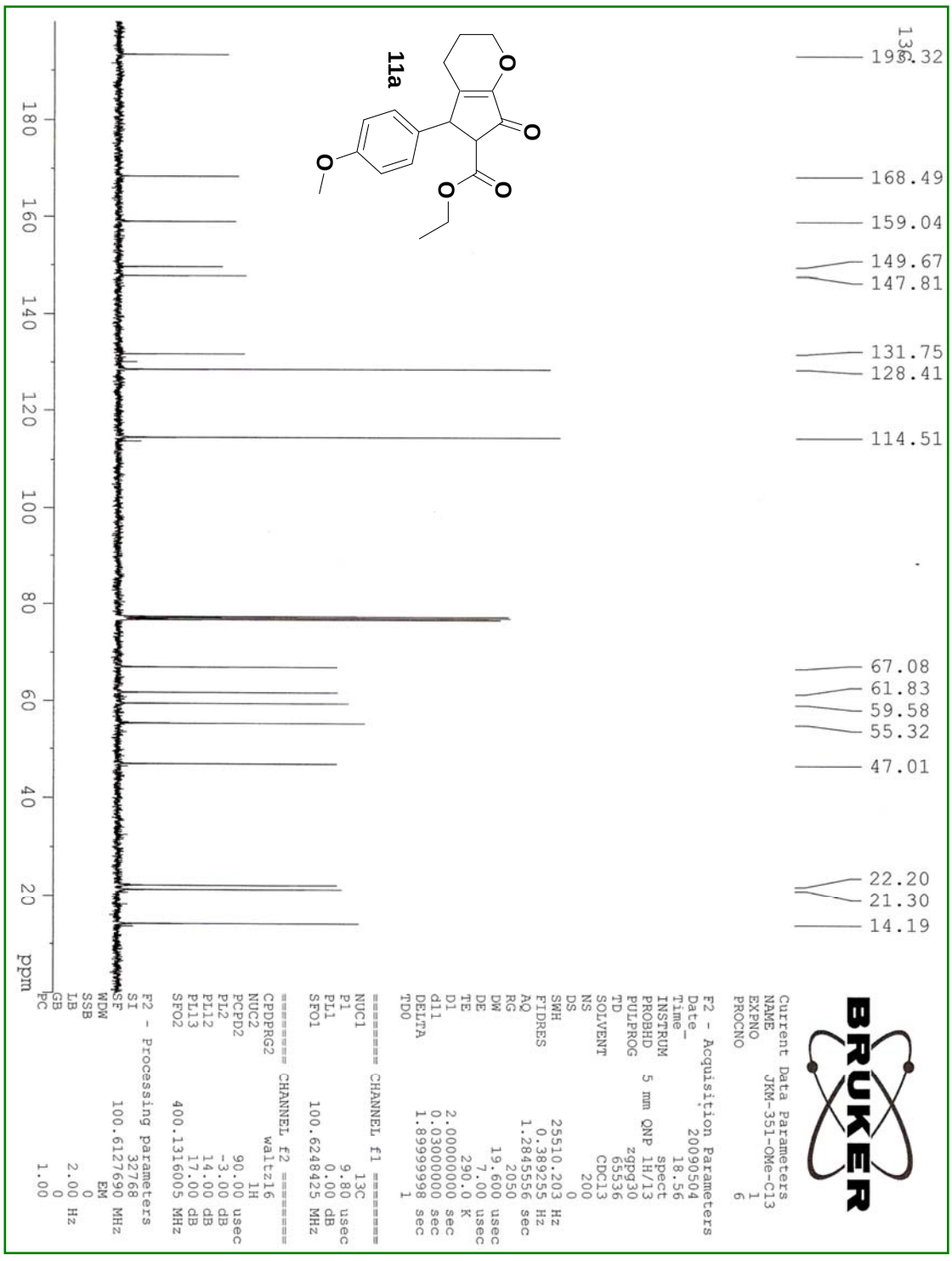

%TRANSMITTANCE
14.20
106.9
4000
3000
2000
1000
400.0
Wavenumbers (cm-1)
3336.25
3081.69
3064.33
3025.76
1708.62
1650.77
1600.63
1494.56
1450.21
1396.21
1388.50
1292.07
1270.86
1226.50
1193.72
1160.94
1105.01
1081.87
1054.87
1027.87
1000.87
944.949
929.521
902.523
873.596
784.886
794.528
678.820
624.823
576.612
561.184
478.260
OH
O
9a
File Name:
Title:
Date & Time: 08/18/09 10:44:51 AM
Resolution: 4 cm-1
Number of Scans: 10
Gain: 1
Comment:
Peak No. W-N(cm-1) %T
01 3336.25 34.3110
02 3081.69 93.6601
03 3064.33 90.5133
04 3025.76 83.0422
05 1708.62 18.5027
06 1650.77 46.4482
Peak No. W-N(cm-1) %T
07 1600.63 87.6402
08 1494.56 64.9139
09 1450.21 66.8486
10 1396.21 38.9079
11 1388.50 30.1850
12 1292.07 95.3596
Peak No. W-N(cm-1) %T
13 1270.86 87.7656
14 1226.50 54.3874
15 1193.72 48.2444
16 1160.94 82.7216
17 1105.01 46.2320
18 1081.87 78.1811
Peak No. W-N(cm-1)
19 1054.87 8
20 1027.87 8
21 1000.87 9
22 944.949 7
23 929.521 9
24 902.523 8

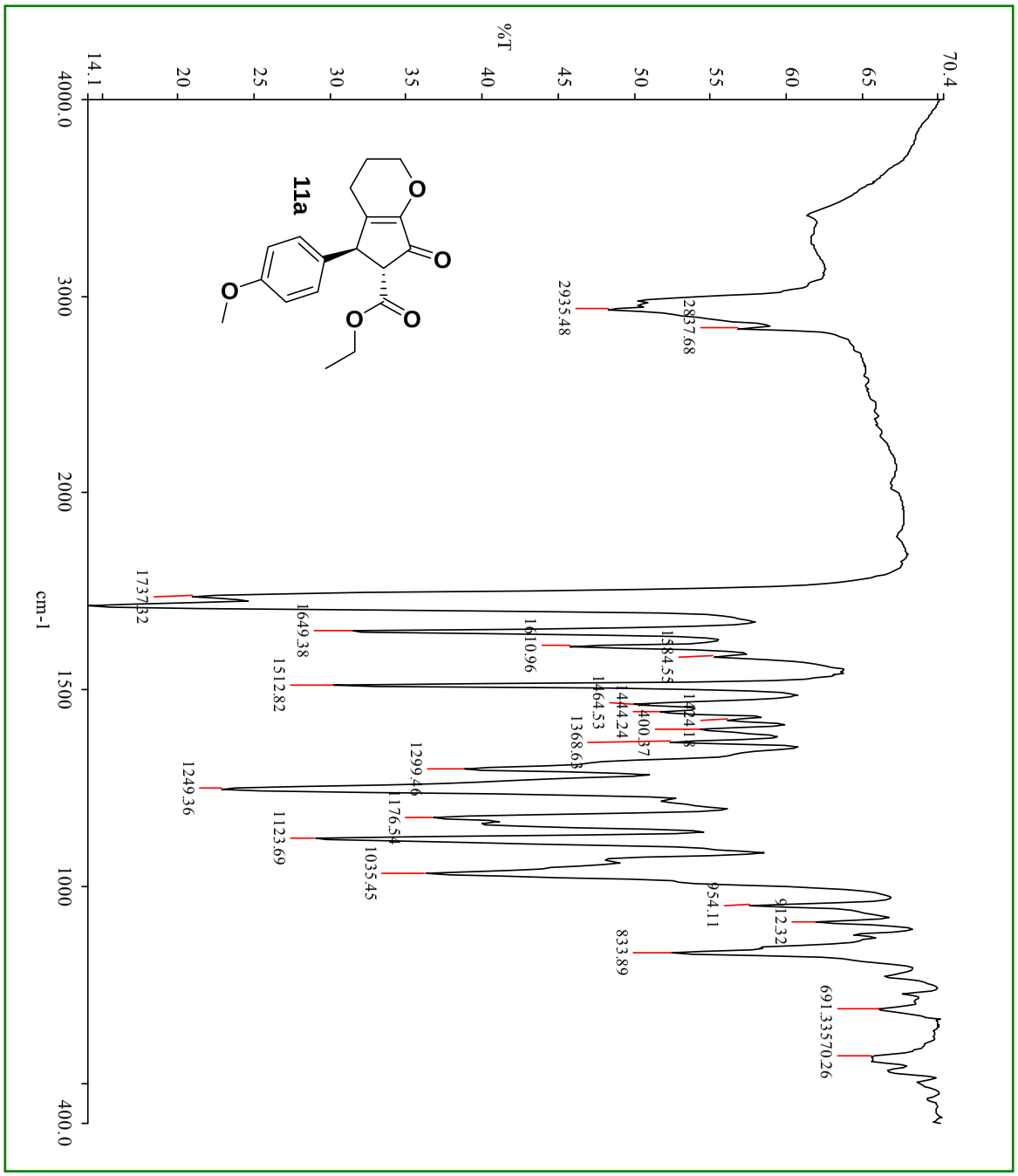

10a
1.9469
0.9025
2.0000
3.1210
1.8646
0.9553
1.0656
1.0326
2.0541
3.0723
ppm
7.3715
7.3442
7.3255
7.3009
7.2833
7.1455
7.1423
7.1252
4.2483
4.2305
4.2130
4.1948
4.1824
4.1662
4.1569
4.1409
4.1231
3.3056
2.2483
2.2323
2.2149
2.2005
2.1853
2.1318
2.1165
2.1013
2.0685
2.0536
2.0061
1.9962
1.9863
1.9706
1.9611
1.9544
1.9451
1.9345
1.9250
1.9181
1.9089
1.9013
1.8921
1.2977
1.2797
1.2620
BRUKER
Current Data Parameters
NAME JKM-343-final
EXPNO 1
PROCNO 6
F2 - Acquisition Parameters
Date_ 20090427
Time_ 16.34
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 5
DS 0
SWH 6393.862 Hz
FIDRES 0.195125 Hz
AQ 2.5625076 sec
RG 322
DW 78.200 usec
DE 6.00 usec
TE 288.9 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.05 usec
PL1 -3.00 dB
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300094 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

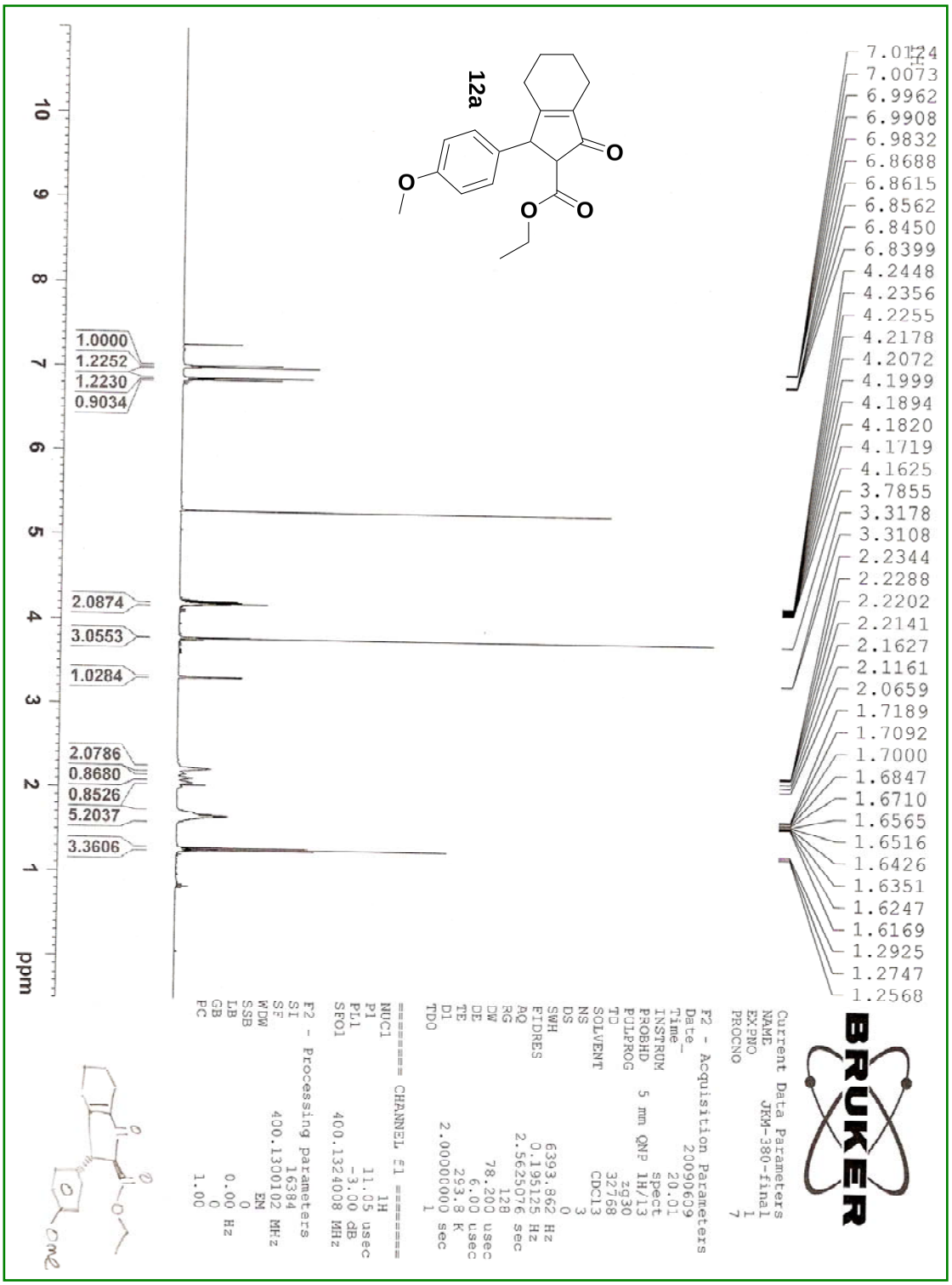

10a
193.15
168.34
149.80
147.45
139.89
129.16
127.65
127.37
67.06
61.88
59.31
47.66
22.21
21.26
14.16
ppm
Current Data Parameters
NAME JKM-343-final-C13
EXPNO 1
PROCNO 6
F2 - Acquisition Parameters
Date_ 20090427
Time 17.01
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 500
DS 0
SWH 25510.203 Hz
FIDRES 0.389255 Hz
AQ 1.2845556 sec
RG 2050
DW 19.600 usec
DE 7.00 usec
TE 289.9 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.80 usec
PL1 0.00 dB
SFO1 100.6248425 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -3.00 dB
PL12 14.00 dB
PL13 17.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127733 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00

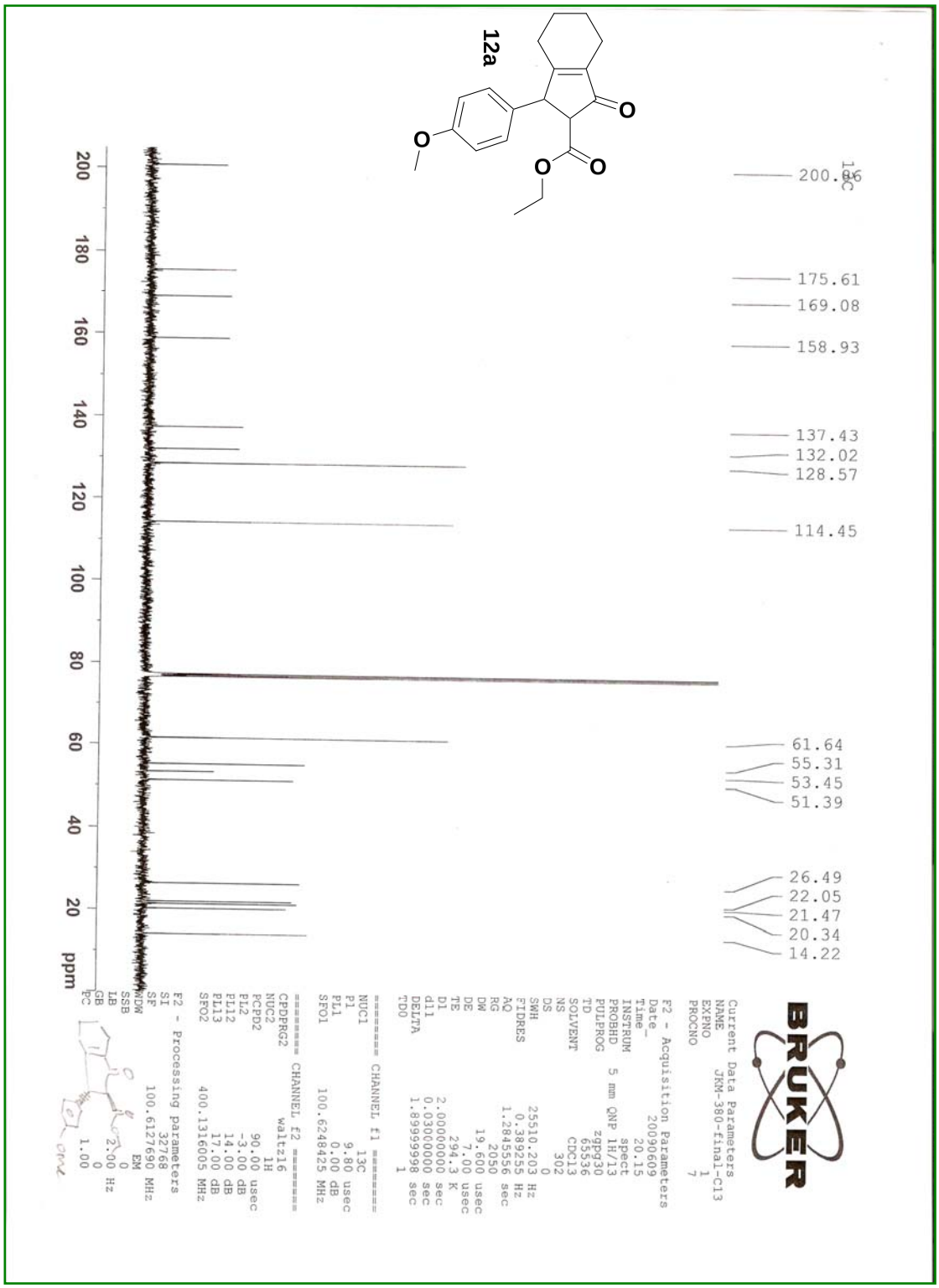

%T
cm-1
10a
3650.28
2979.93
2918.55
2346.66
1738.52
1649.52
1602.92
1496.07
1459.28
1444.37
1424.14
1371.11
1326.58
1295.83
1247.16
1222.24
1171.18
1129.52
1051.54
1015.72
953.73
928.36
857.89
834.40
792.70
781.33
705.36
636.01
564.44
497.44
467.30

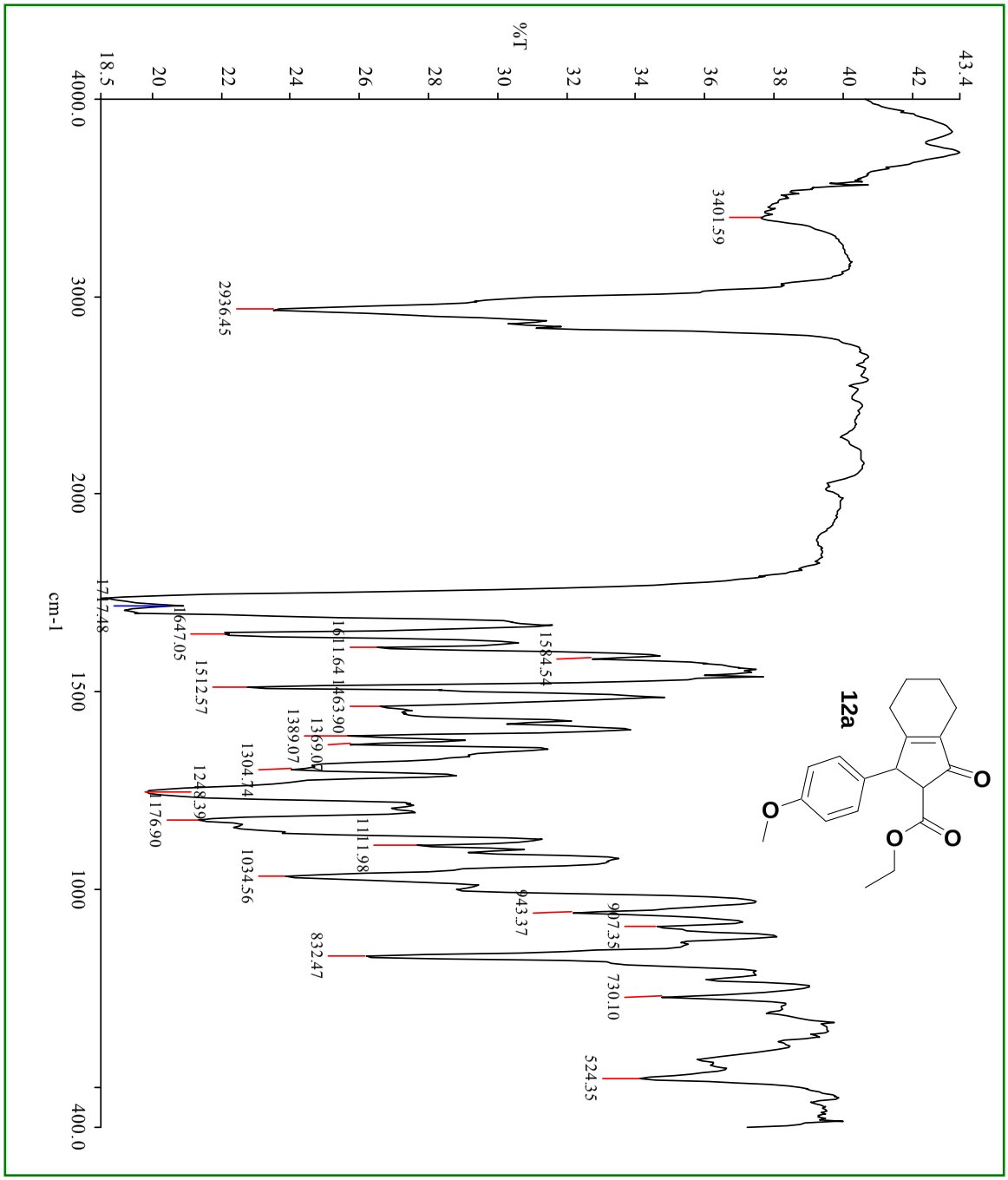

11a
7.0423
7.0209
6.8602
6.8389
5.2777
4.2197
4.2020
4.1842
4.1665
4.1412
4.1268
4.1111
3.7735
3.2427
3.2386
2.2285
2.2129
2.1970
2.1805
2.1652
2.1497
2.1141
2.0989
2.0837
2.0510
2.0356
2.0195
1.9803
1.9706
1.9610
1.9450
1.9351
1.9291
1.9185
1.9099
1.9003
1.8935
1.8840
1.8660
1.8583
1.8484
1.2726
1.2548
1.2369
2.1188
2.0000
2.3010
2.9121
3.0131
0.9827
1.2173
1.2032
2.4962
3.5310
ppm
BRUKER
Current Data Parameters
NAME JKM-351-OMe
EXPNO 1
PROCNO 6
F2 - Acquisition Parameters
Date_ 20090504
Time_ 18.37
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 3
DS 0
SWH 6393.862 Hz
FIDRES 0.195125 Hz
AQ 2.5625076 sec
RG 64
DW 78.200 usec
DE 6.00 usec
TE 289.6 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.05 usec
PL1 -3.00 dB
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300091 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

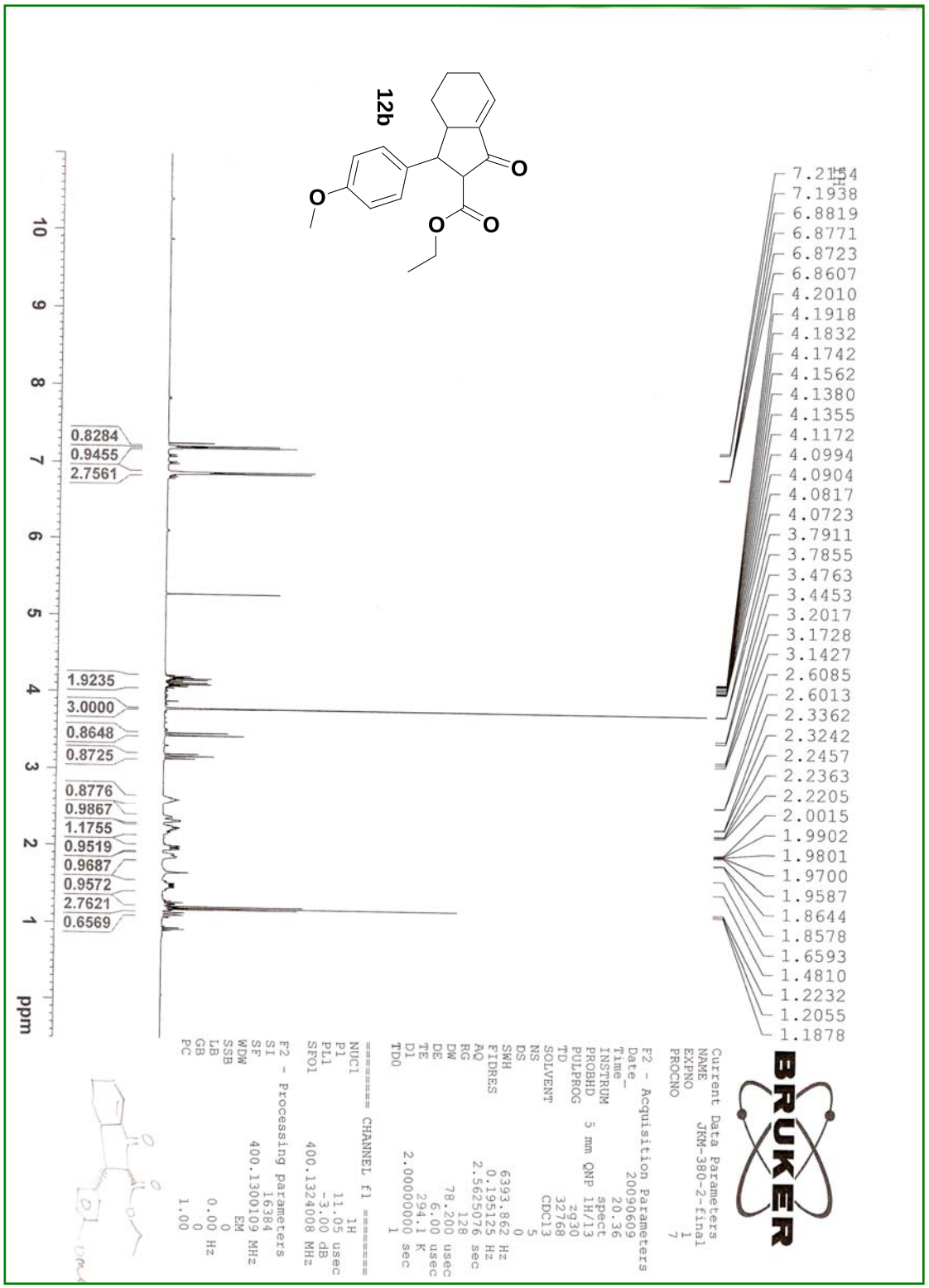

11a
196.32
168.49
159.04
149.67
147.81
131.75
128.41
114.51
67.08
61.83
59.58
55.32
47.01
22.20
21.30
14.19
ppm
Current Data Parameters
NAME JKM-351-OMe-C13
EXPNO 1
PROCNO 6
F2 - Acquisition Parameters
Date_ 20090504
Time_ 18.56
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 200
DS 0
SWH 25510.203 Hz
FIDRES 0.389255 Hz
AQ 1.2845556 sec
RG 2050
DW 19.600 usec
DE 7.00 usec
TE 290.0 K
D1 2.00000000 sec
D11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.80 usec
PL1 0.00 dB
SFO1 100.6248425 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -3.00 dB
PL12 14.00 dB
PL13 17.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00

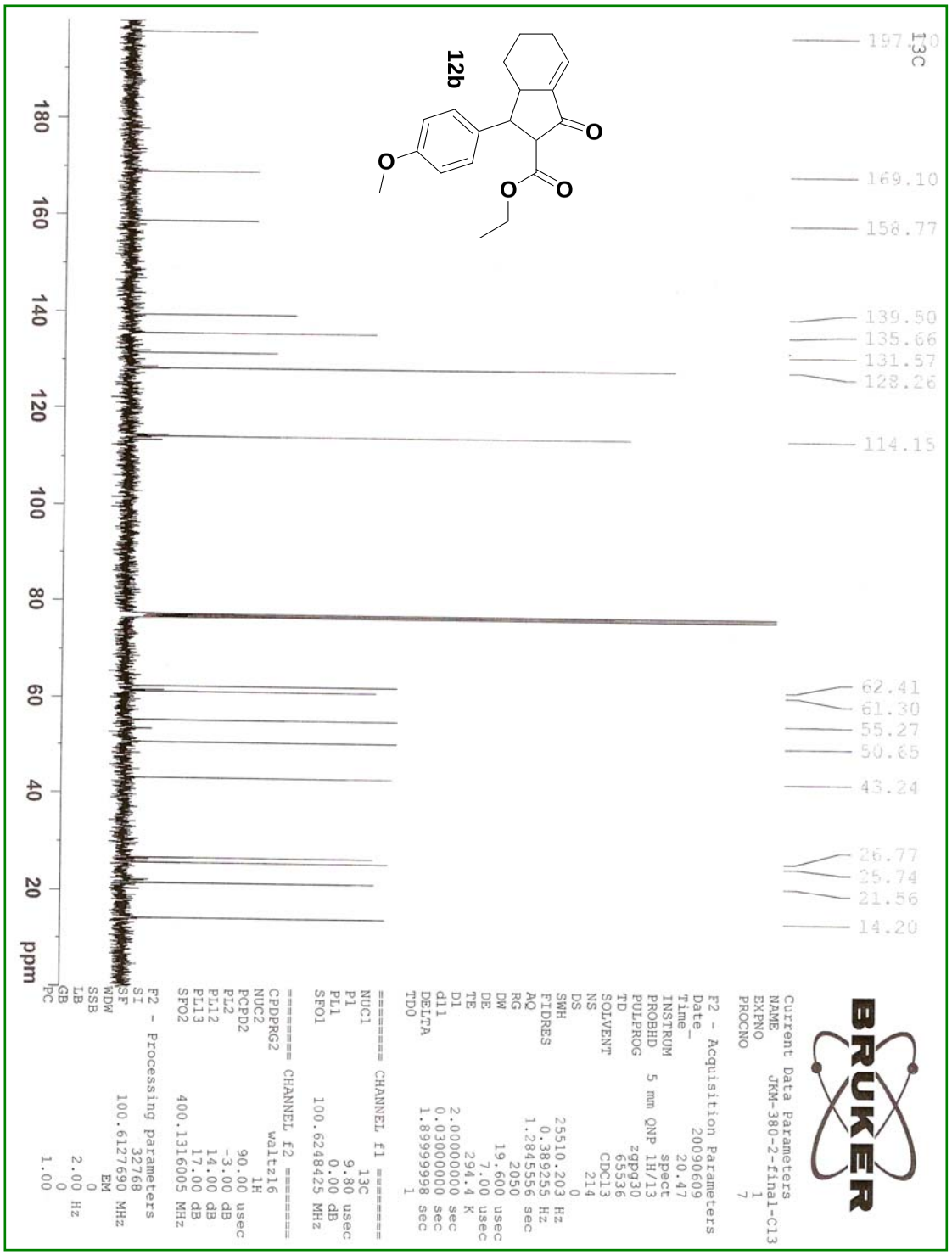

11a
%T
cm-1
70.4
65
60
55
50
45
40
35
30
25
20
14.1
4000.0
3000
2000
1500
1000
400.0
2935.48
2837.68
1737.32
1649.38
1610.96
1584.55
1512.82
1464.53
1444.24
1400.57
1368.63
1299.46
1249.36
1176.54
1123.69
1035.45
954.11
912.32
833.89
691.33
570.26

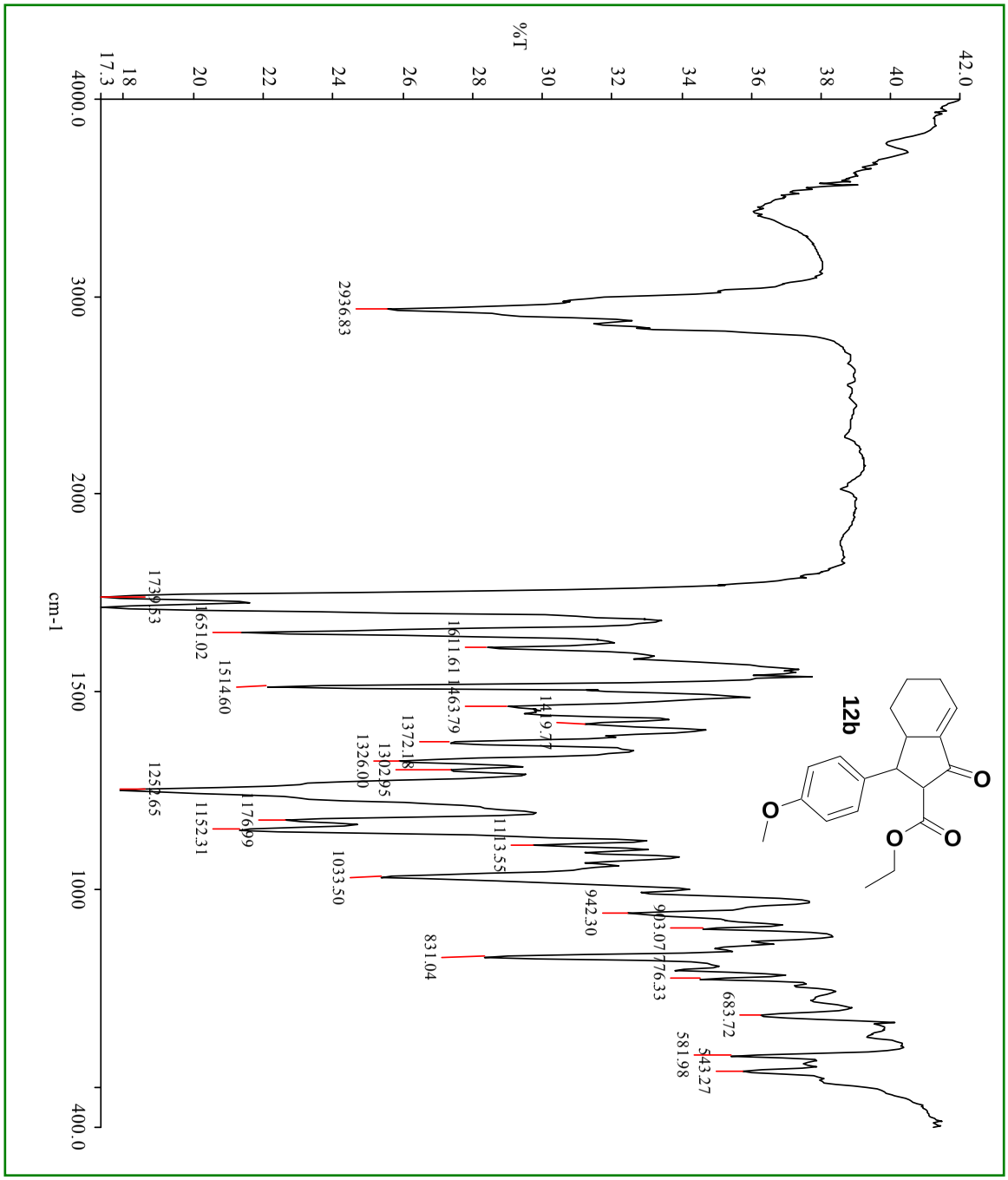

12a
O
O
O
O
7.0124
7.0073
6.9962
6.9908
6.9832
6.8688
6.8615
6.8562
6.8450
6.8399
4.2448
4.2356
4.2255
4.2178
4.2072
4.1999
4.1894
4.1820
4.1719
4.1625
3.7855
3.3178
3.3108
2.2344
2.2288
2.2202
2.2141
2.1627
2.1161
2.0659
1.7189
1.7092
1.7000
1.6847
1.6710
1.6565
1.6516
1.6426
1.6351
1.6247
1.6169
1.2925
1.2747
1.2568
1.0000
1.2252
1.2230
0.9034
2.0874
3.0553
1.0284
2.0786
0.8680
0.8526
5.2037
3.3606
ppm
BRUKER
Current Data Parameters
NAME JKM-380-final
EXPNO 1
PROCNO 7
F2 - Acquisition Parameters
Date_ 20090609
Time_ 20.01
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 3
DS 0
SWH 6393.862 Hz
FIDRES 0.195125 Hz
AQ 2.5625076 sec
RG 128
DW 78.200 usec
DE 6.00 usec
TE 293.8 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.05 usec
PL1 -3.00 dB
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300102 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

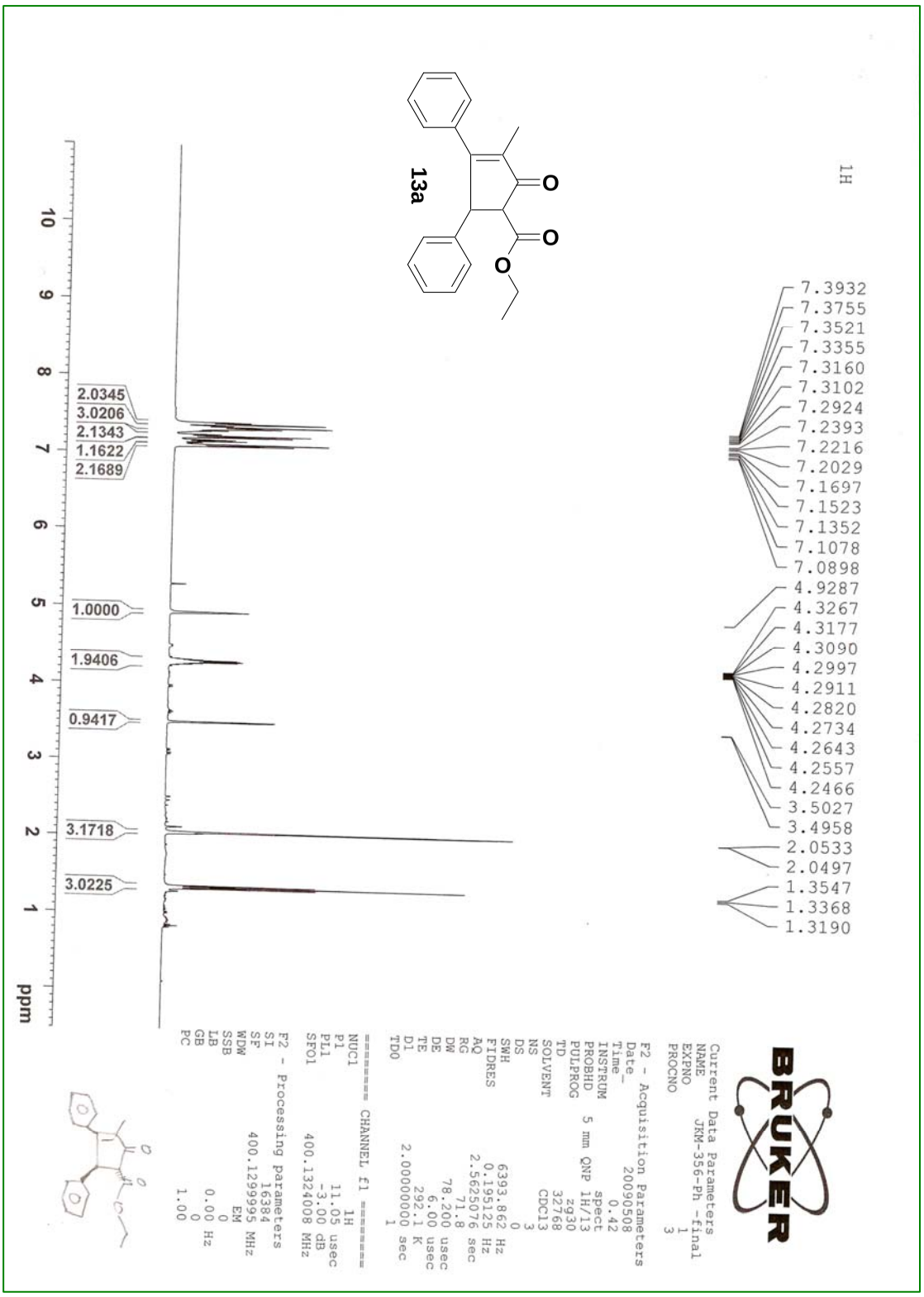

12a
O
O
O
O
200.86
175.61
169.08
158.93
137.43
132.02
128.57
114.45
61.64
55.31
53.45
51.39
26.49
22.05
21.47
20.34
14.22
200
180
160
140
120
100
80
60
40
20
ppm
BRUKER

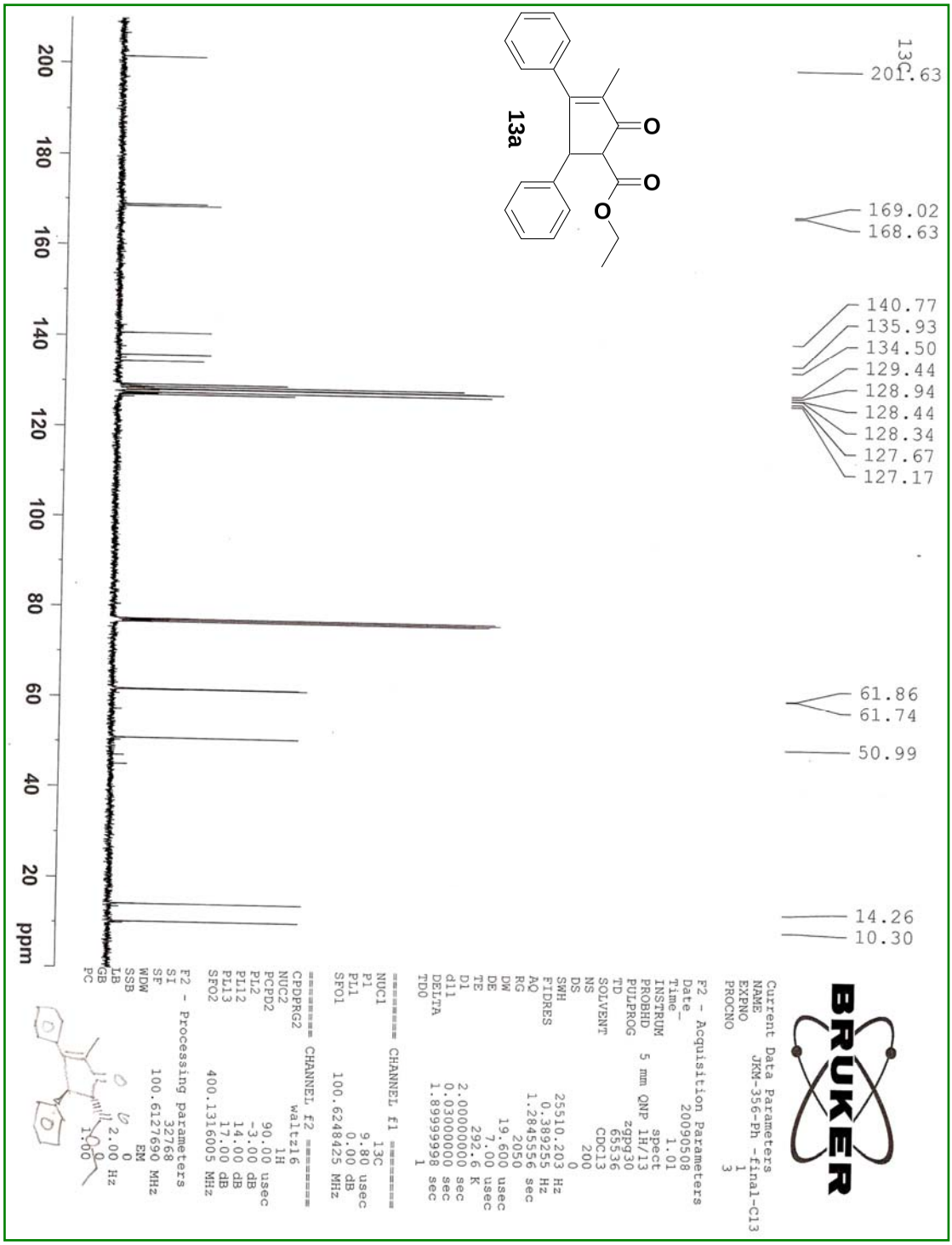

12a
%T
cm-1
43.4
42
40
38
36
34
32
30
28
26
24
22
20
18.5
4000.0
3000
2000
1500
1000
400.0
3401.59
2936.45
1717.48
1647.05
1611.64
1584.54
1512.57
1463.90
1389.07
1369.07
1304.74
1248.39
1176.90
1111.98
1034.56
943.37
907.35
832.47
730.10
524.35

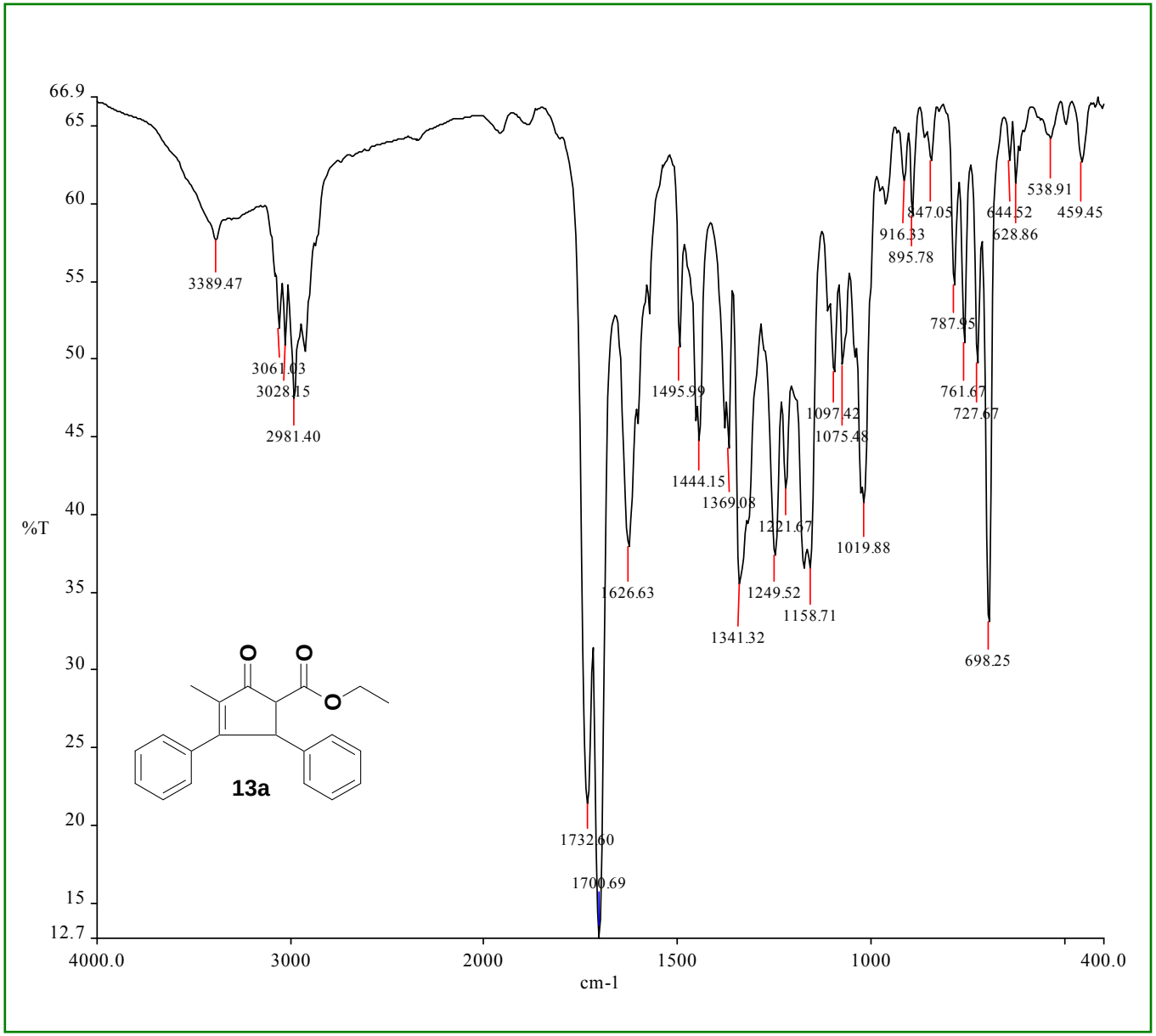

12b
O
O
O
O
7.2154
7.1938
6.8819
6.8771
6.8723
6.8607
4.2010
4.1918
4.1832
4.1742
4.1562
4.1380
4.1355
4.1172
4.0994
4.0904
4.0817
4.0723
3.7911
3.7855
3.4763
3.4453
3.2017
3.1728
3.1427
2.6085
2.6013
2.3362
2.3242
2.2457
2.2363
2.2205
2.0015
1.9902
1.9801
1.9700
1.9587
1.8644
1.8578
1.6593
1.4810
1.2232
1.2055
1.1878
0.8284
0.9455
2.7561
1.9235
3.0000
0.8648
0.8725
0.8776
0.9867
1.1755
0.9519
0.9687
0.9572
2.7621
0.6569
ppm
Current Data Parameters
NAME JKM-380-2-final
EXPNO 1
PROCNO 7
F2 - Acquisition Parameters
Date_ 20090609
Time_ 20.36
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 5
DS 0
SWH 6393.862 Hz
FIDRES 0.195125 Hz
AQ 2.5625076 sec
RG 128
DW 78.200 usec
DE 6.00 usec
TE 294.1 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.05 usec
PL1 -3.00 dB
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300109 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
BRUKER

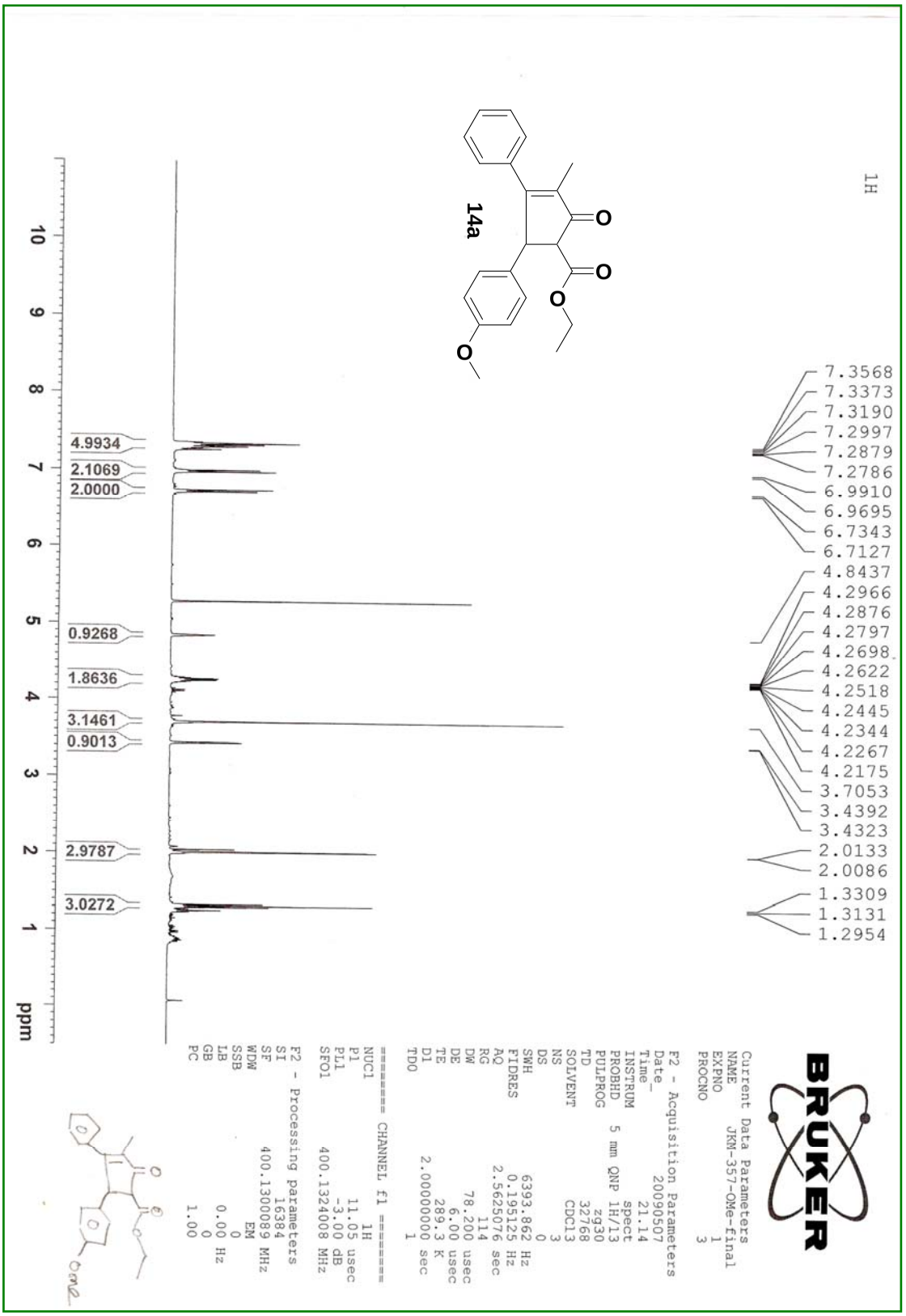

13C
197.10
169.10
158.77
139.50
135.66
131.57
128.26
114.15
62.41
61.30
55.27
50.65
43.24
26.77
25.74
21.56
14.20
12b
O
O
O
O
180
160
140
120
100
80
60
40
20
ppm
BRUKER
Current Data Parameters
NAME JKM-380-2-final-C13
EXPNO 1
PROCNO 7
F2 - Acquisition Parameters
Date_ 20090609
Time_ 20.47
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 214
DS 0
SWH 25510.203 Hz
FIDRES 0.389255 Hz
AQ 1.2845556 sec
RG 2050
DW 19.600 usec
DE 7.00 usec
TE 294.4 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.80 usec
PL1 0.00 dB
SFO1 100.6248425 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -3.00 dB
PL12 14.00 dB
PL13 17.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00

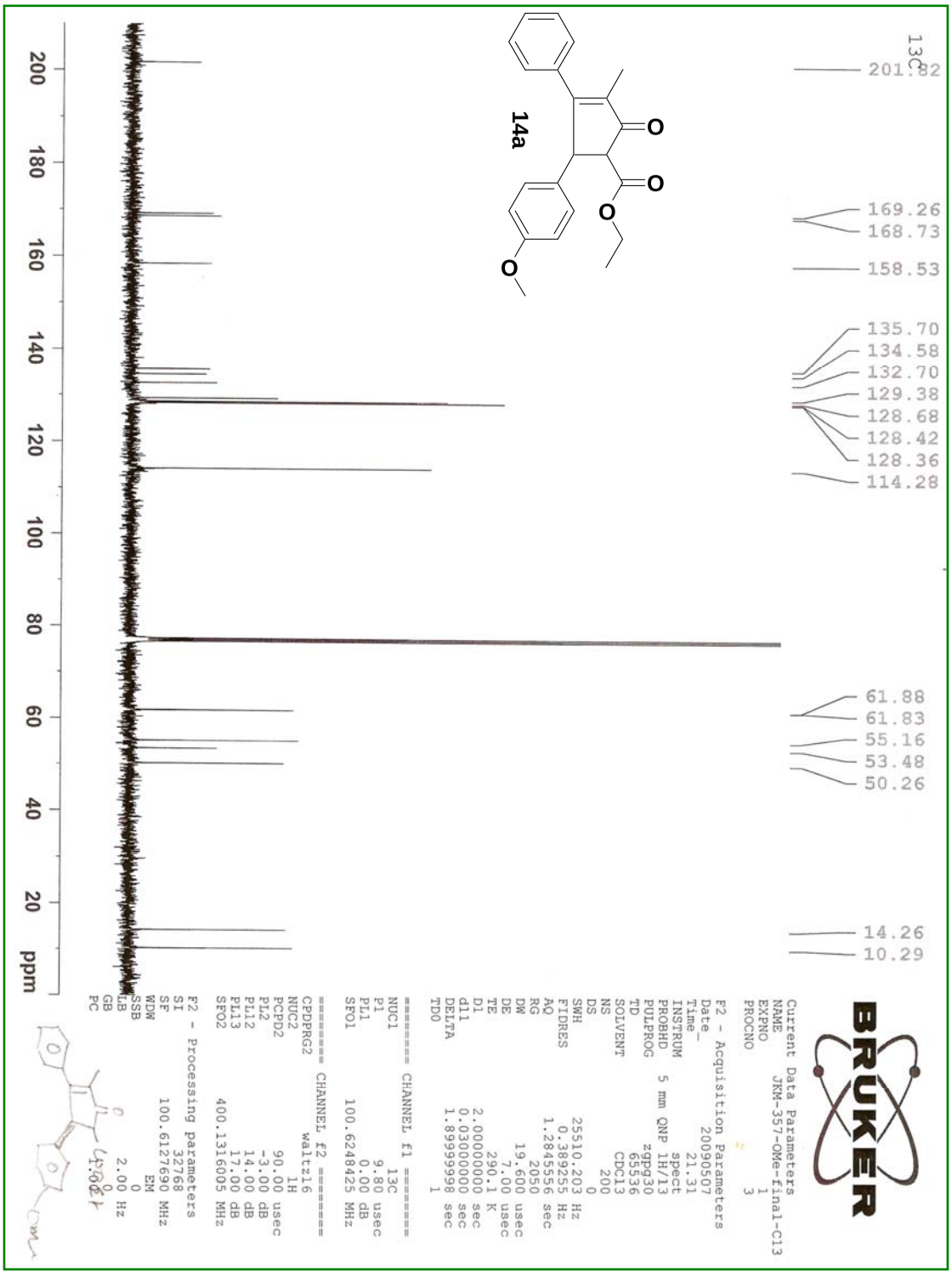

12b
%T
cm-1
42.0
40
38
36
34
32
30
28
26
24
22
20
18
17.3
4000.0
3000
2000
1500
1000
400.0
2936.83
1730.53
1651.02
1611.61
1514.60
1463.79
1419.77
1372.18
1302.95
1326.00
1252.65
1176.99
1152.31
1113.55
1033.50
942.30
903.07
876.33
831.04
683.72
581.98
543.27
O
O
O
O

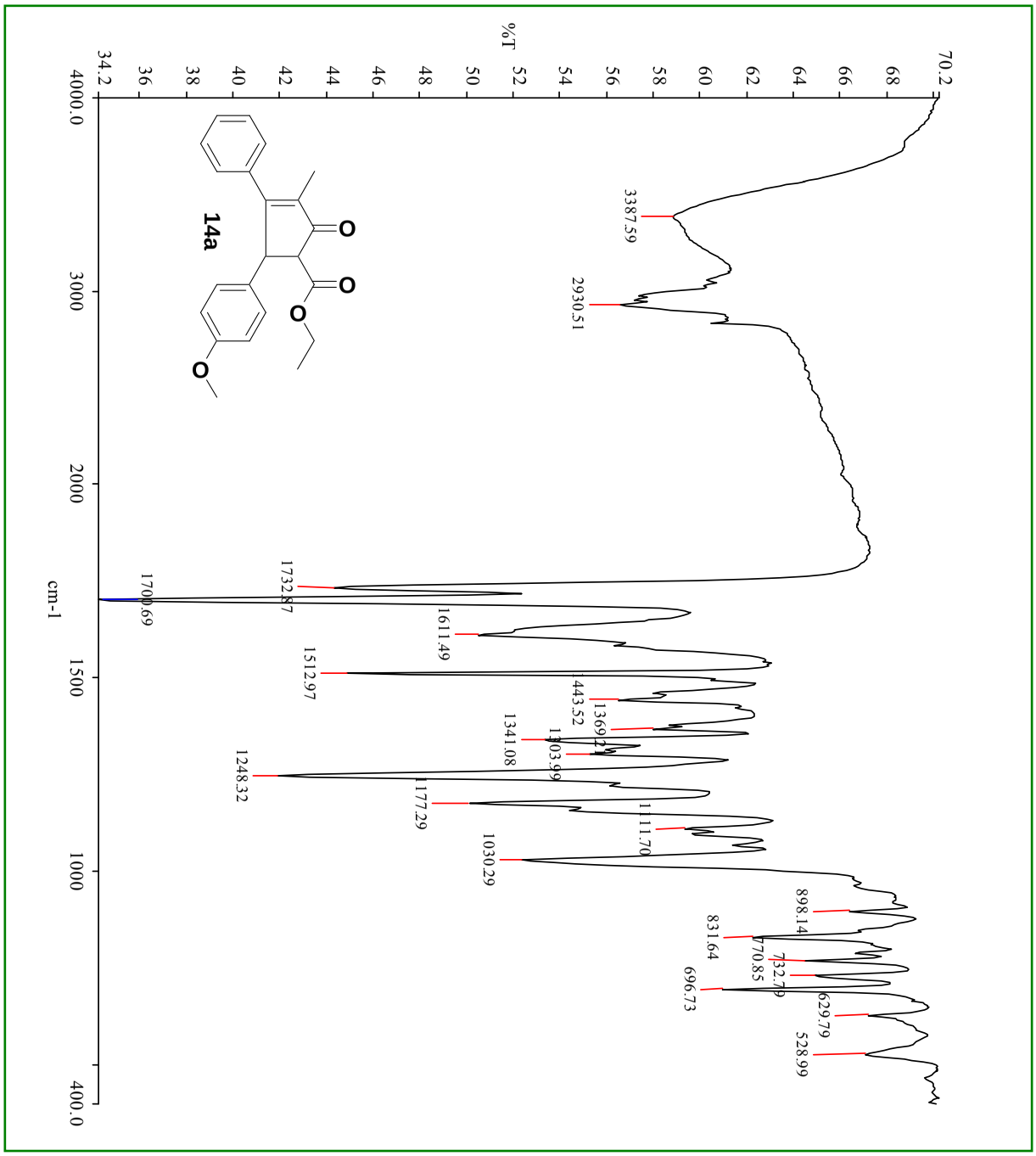

1H
13a
O
O
O
7.3932
7.3755
7.3521
7.3355
7.3160
7.3102
7.2924
7.2393
7.2216
7.2029
7.1697
7.1523
7.1352
7.1078
7.0898
4.9287
4.3267
4.3177
4.3090
4.2997
4.2911
4.2820
4.2734
4.2643
4.2557
4.2466
3.5027
3.4958
2.0533
2.0497
1.3547
1.3368
1.3190
2.0345
3.0206
2.1343
1.1622
2.1689
1.0000
1.9406
0.9417
3.1718
3.0225
ppm
BRUKER
Current Data Parameters
NAME JKM-356-Ph -final
EXPNO 1
PROCNO 3
F2 - Acquisition Parameters
Date_ 20090508
Time_ 0.42
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDC13
NS 3
DS 0
SWH 6393.862 Hz
FIDRES 0.195125 Hz
AQ 2.5625076 sec
RG 71.8
DW 78.200 usec
DE 6.00 usec
TE 292.1 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.05 usec
PL1 -3.00 dB
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 16384
SF 400.1299995 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

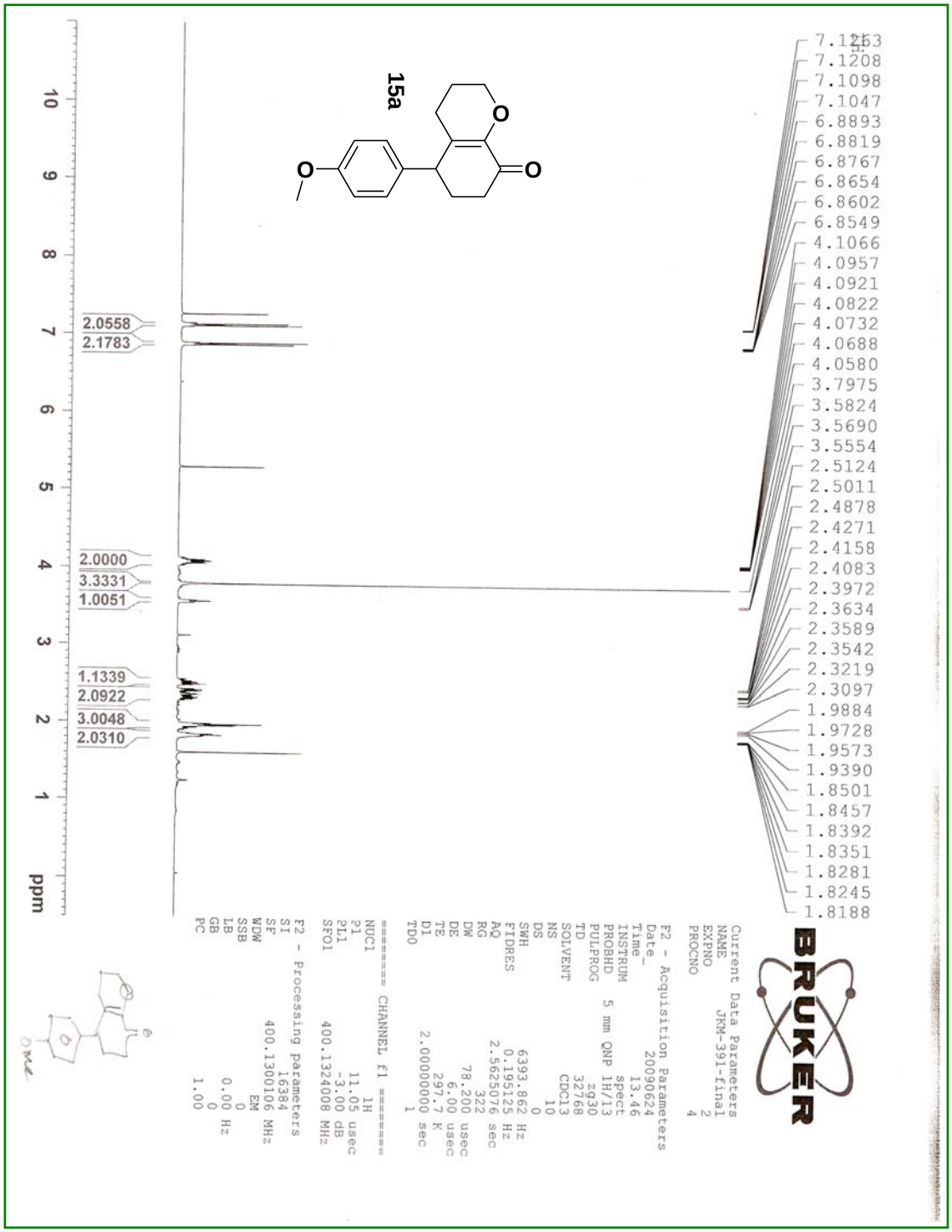

13C
201.63
169.02
168.63
140.77
135.93
134.50
129.44
128.94
128.44
128.34
127.67
127.17
61.86
61.74
50.99
14.26
10.30
13a
O
O
O
200
180
160
140
120
100
80
60
40
20
ppm
BRUKER
Current Data Parameters
NAME JKM-356-Ph -final-C13
EXPNO 1
PROCNO 3
F2 - Acquisition Parameters
Date_ 20090508
Time_ 1.01
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 200
DS 0
SWH 25510.203 Hz
FIDRES 0.389255 Hz
AQ 1.2845556 sec
RG 2050
DW 19.600 usec
DE 7.00 usec
TE 292.6 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.80 usec
PL1 0.00 dB
SFO1 100.6248425 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -3.00 dB
PL12 14.00 dB
PL13 17.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00

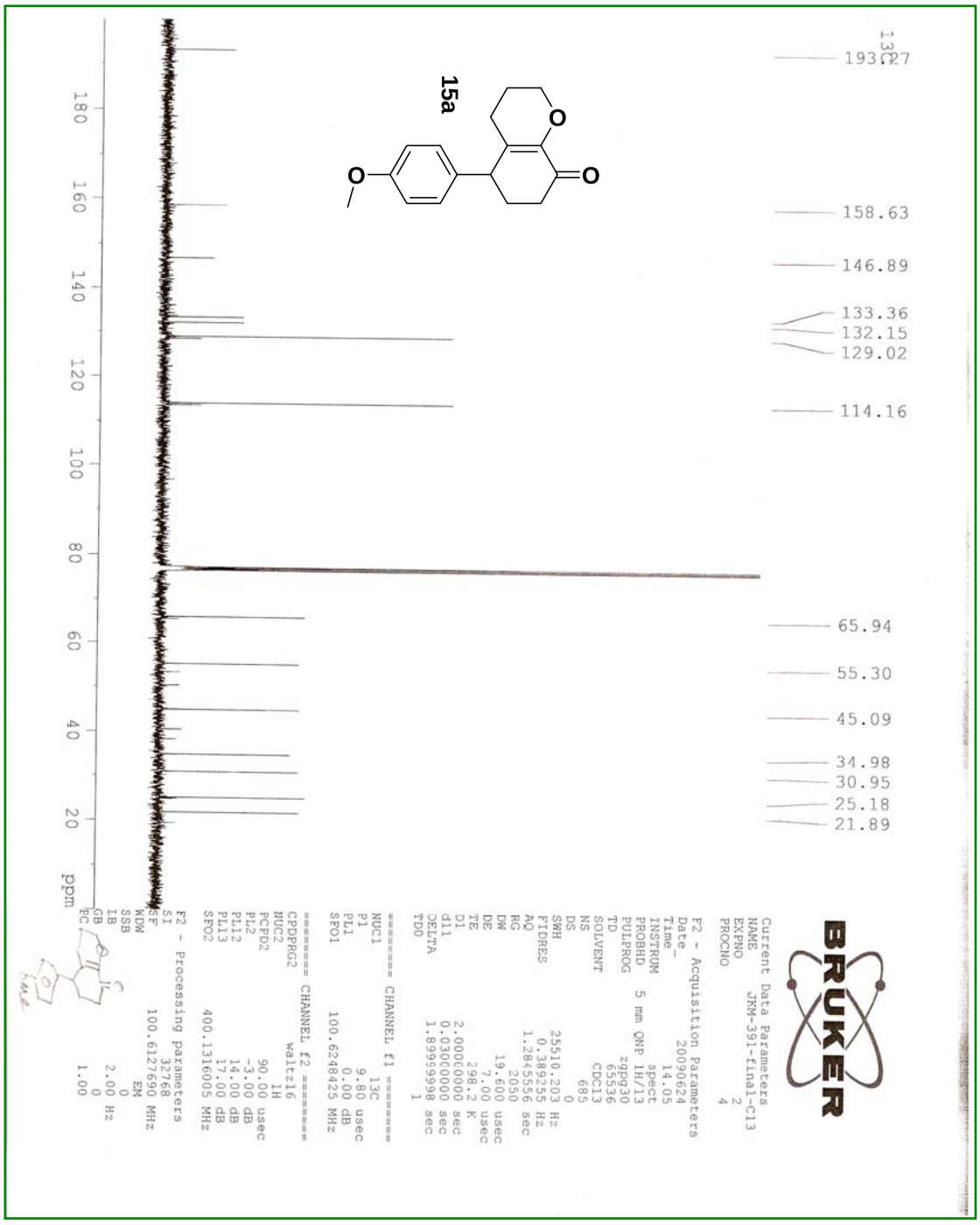


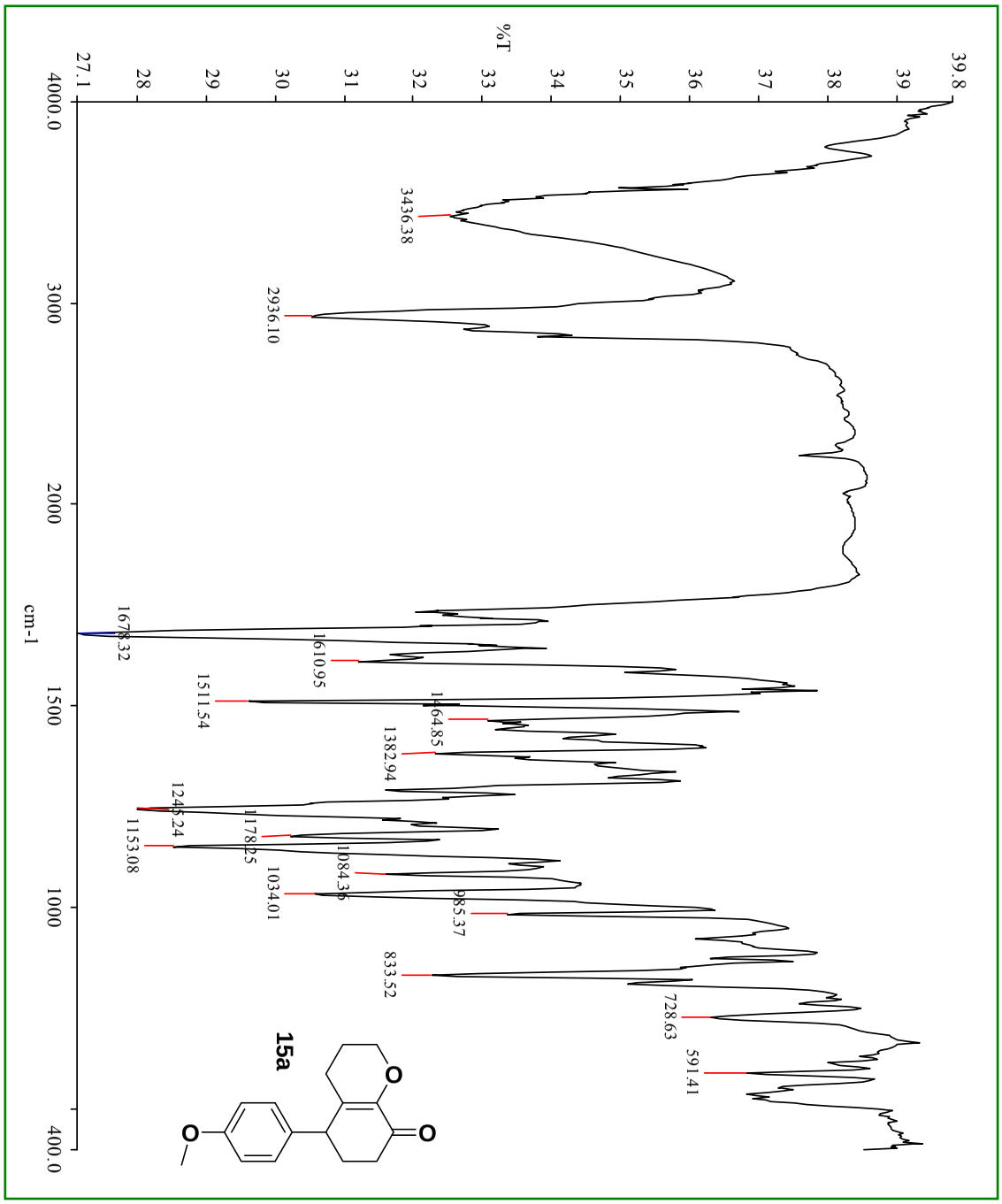

1H
14a
7.3568
7.3373
7.3190
7.2997
7.2879
7.2786
6.9910
6.9695
6.7343
6.7127
4.8437
4.2966
4.2876
4.2797
4.2698
4.2622
4.2518
4.2445
4.2344
4.2267
4.2175
3.7053
3.4392
3.4323
2.0133
2.0086
1.3309
1.3131
1.2954
4.9934
2.1069
2.0000
0.9268
1.8636
3.1461
0.9013
2.9787
3.0272
ppm
BRUKER
Current Data Parameters
NAME JKM-357-OMe-final
EXPNO 1
PROCNO 3
F2 - Acquisition Parameters
Date_ 20090507
Time_ 21.14
INSTRUM spect
PROBHD 5 mm QNP 1h/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 3
DS 0
SWH 6393.862 Hz
FIDRES 0.195125 Hz
AQ 2.5625076 sec
RG 114
DW 78.200 usec
DE 6.00 usec
TE 289.3 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.05 usec
PL1 -3.00 dB
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300089 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

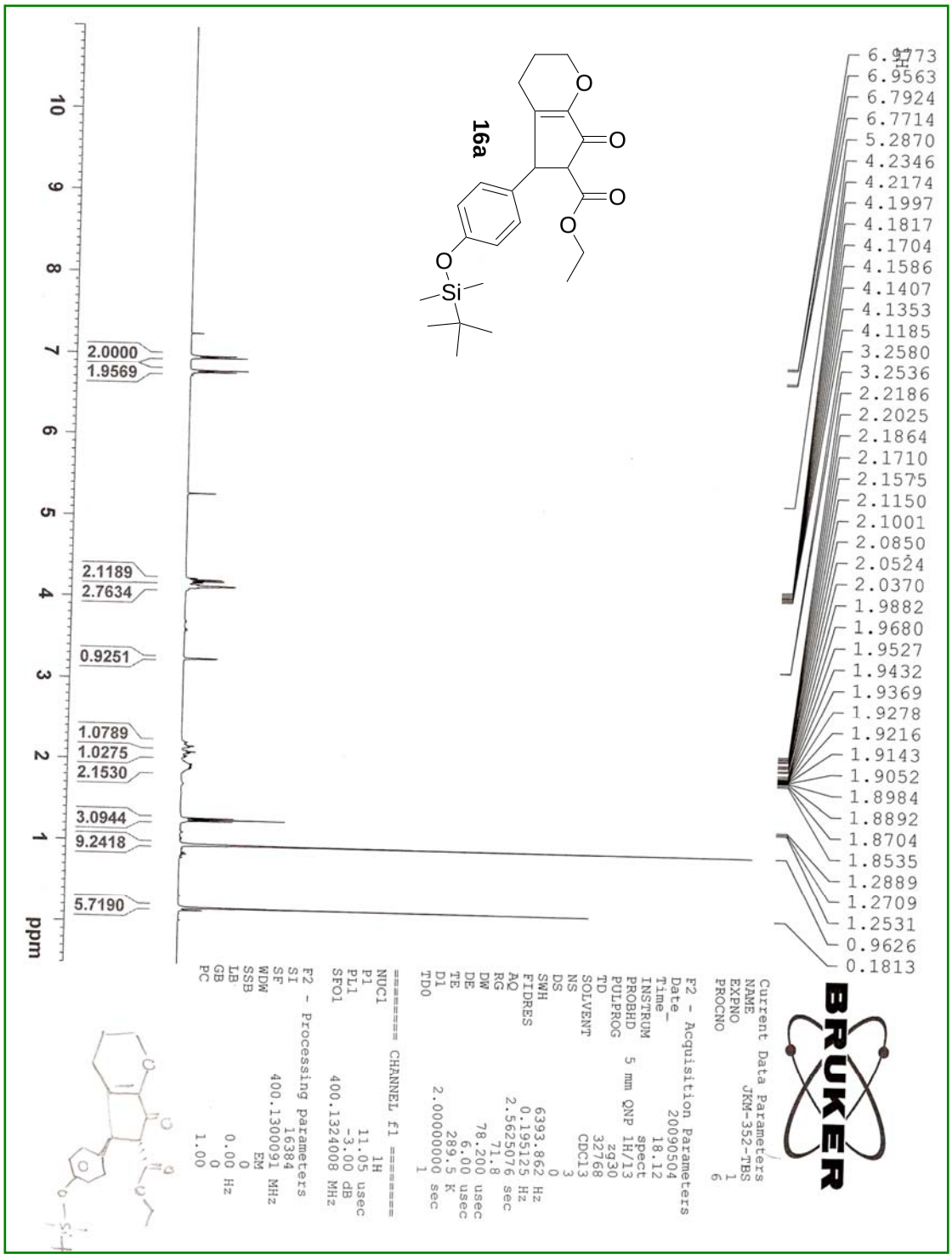

13C
201.82
169.26
168.73
158.53
135.70
134.58
132.70
129.38
128.68
128.42
128.36
114.28
61.88
61.83
55.16
53.48
50.26
14.26
10.29
14a
O
O
O
O
200
180
160
140
120
100
80
60
40
20
ppm
BRUKER
Current Data Parameters
NAME JKM-357-OMe-final-C13
EXPNO 1
PROCNO 3
F2 - Acquisition Parameters
Date_ 20090507
Time 21.31
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 200
DS 0
SWH 25510.203 Hz
FIDRES 0.389255 Hz
AQ 1.2845556 sec
RG 2050
DW 19.600 usec
DE 7.00 usec
TE 290.1 K
D1 2.00000000 sec
D11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.80 usec
PL1 0.00 dB
SFO1 100.6248425 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -3.00 dB
PL12 14.00 dB
PL13 17.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00

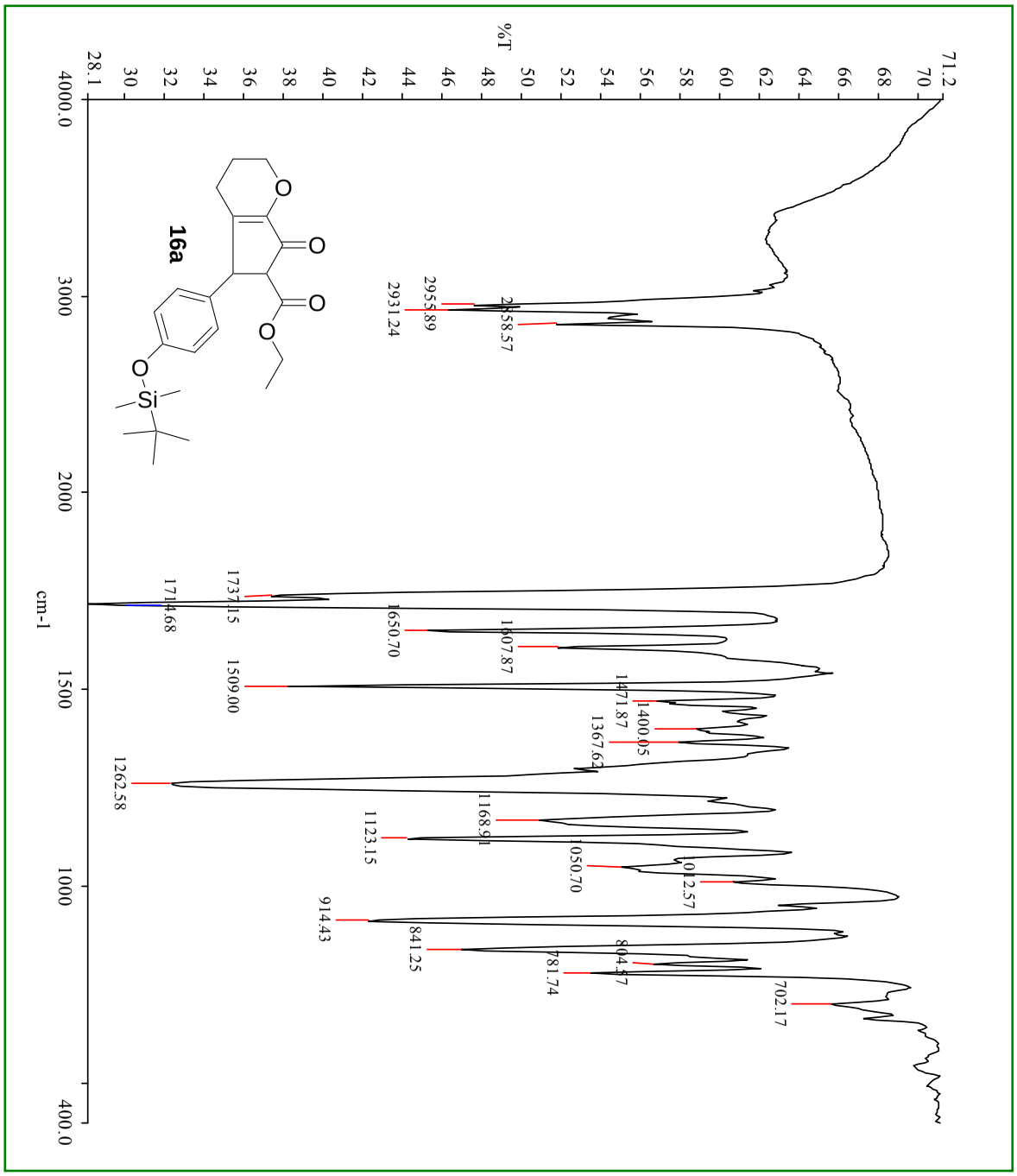

%T
cm-1
14a
3387.59
2930.51
1732.87
1700.69
1611.49
1512.97
1443.52
1369.21
1341.08
1303.99
1248.32
1177.29
1111.70
1030.29
898.14
831.64
770.85
732.79
696.73
629.79
528.99

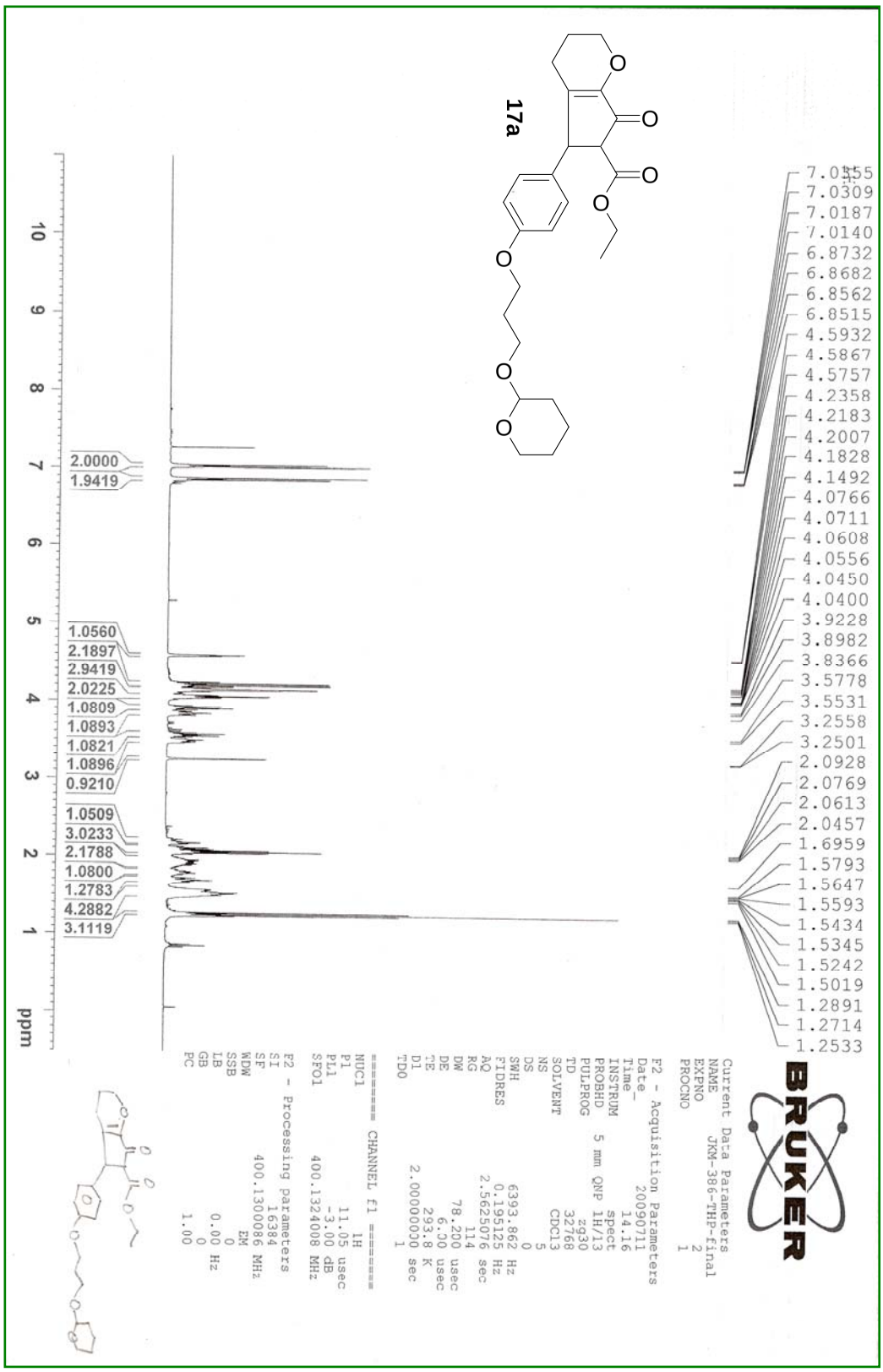

15a
O
O
O
7.1263
7.1208
7.1098
7.1047
6.8893
6.8819
6.8767
6.8654
6.8602
6.8549
4.1066
4.0957
4.0921
4.0822
4.0732
4.0688
4.0580
3.7975
3.5824
3.5690
3.5554
2.5124
2.5011
2.4878
2.4271
2.4158
2.4083
2.3972
2.3634
2.3589
2.3542
2.3219
2.3097
1.9884
1.9728
1.9573
1.9390
1.8501
1.8457
1.8392
1.8351
1.8281
1.8245
1.8188
2.0558
2.1783
2.0000
3.3331
1.0051
1.1339
2.0922
3.0048
2.0310
ppm
BRUKER
Current Data Parameters
NAME JKM-391-final
EXPNO 2
PROCNO 4
F2 - Acquisition Parameters
Date_ 20090624
Time_ 13.46
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 10
DS 0
SWH 6393.862 Hz
FIDRES 0.195125 Hz
AQ 2.5625076 sec
RG 322
DW 78.200 usec
DE 6.00 usec
TE 297.7 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.05 usec
PL1 -3.00 dB
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300106 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

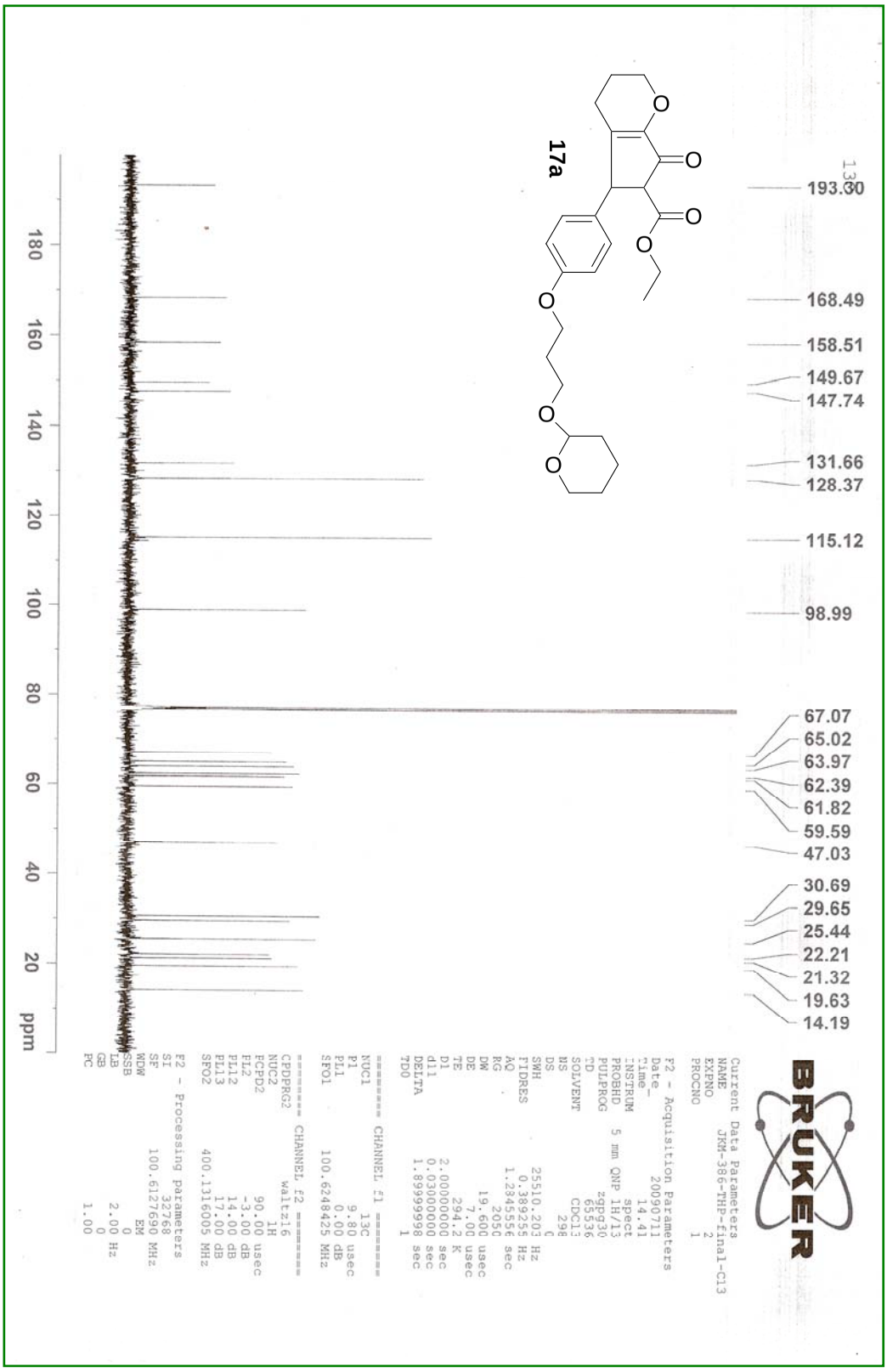

15a
193.27
158.63
146.89
133.36
132.15
129.02
114.16
65.94
55.30
45.09
34.98
30.95
25.18
21.89
ppm
BRUKER
Current Data Parameters
NAME JRM-391-final-C13
EXPNO 2
PROCNO 4
F2 - Acquisition Parameters
Date_ 20090624
Time_ 14.05
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 685
DS 0
SWH 25510.203 Hz
FIDRES 0.389255 Hz
AQ 1.2845556 sec
RG 2050
DW 19.600 usec
DE 7.00 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.80 usec
PL1 0.00 dB
SFO1 100.6248425 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 13.00 dB
PL12 14.00 dB
PL13 17.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00

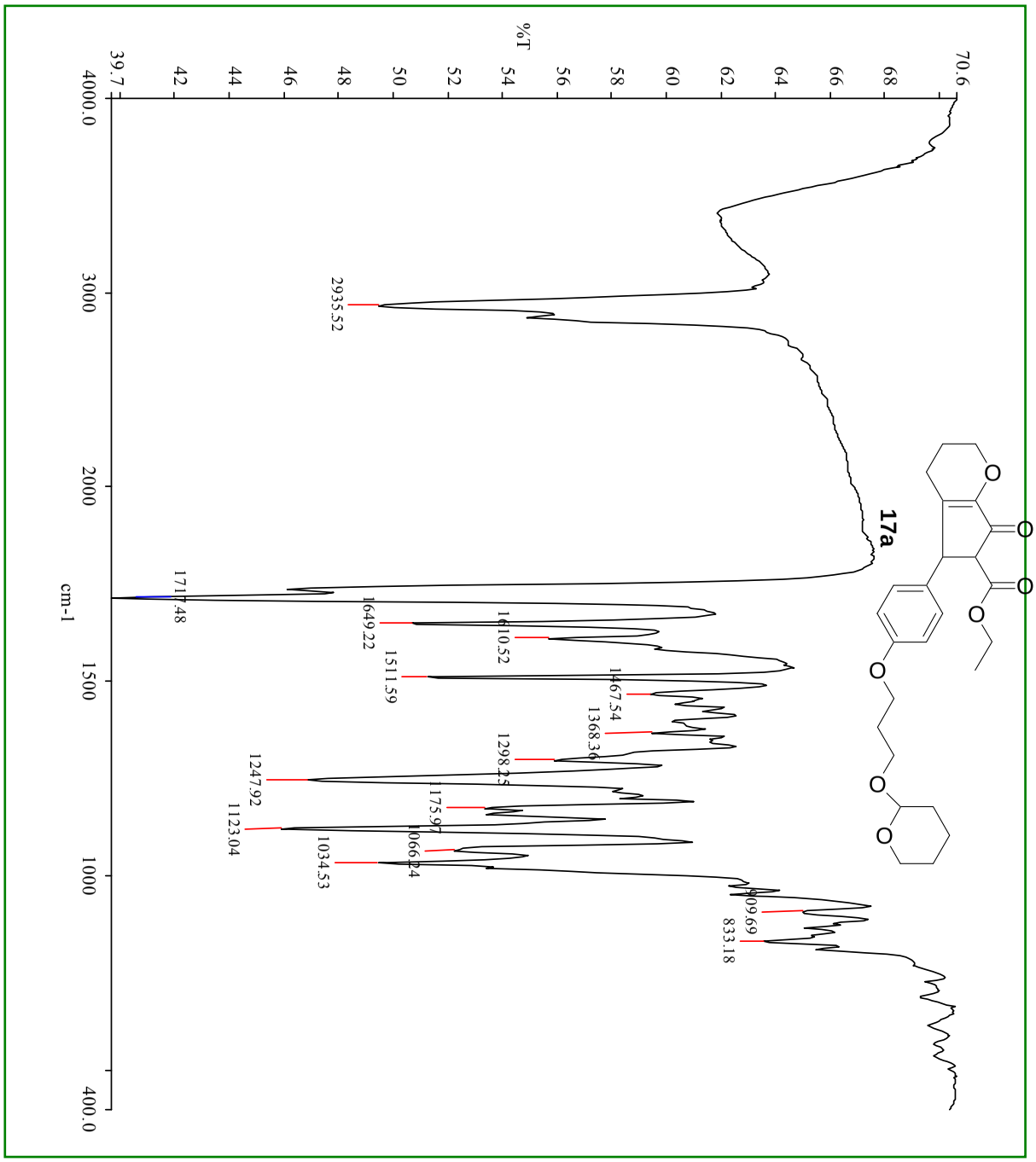

%T
cm-1
39.8
39
38
37
36
35
34
33
32
31
30
29
28
27.1
4000.0
3000
2000
1500
1000
400.0
3436.38
2936.10
1678.32
1610.95
1511.54
1464.85
1382.94
1245.24
1178.25
1153.08
1084.36
1034.01
985.37
833.52
728.63
591.41
15a
O
O
O

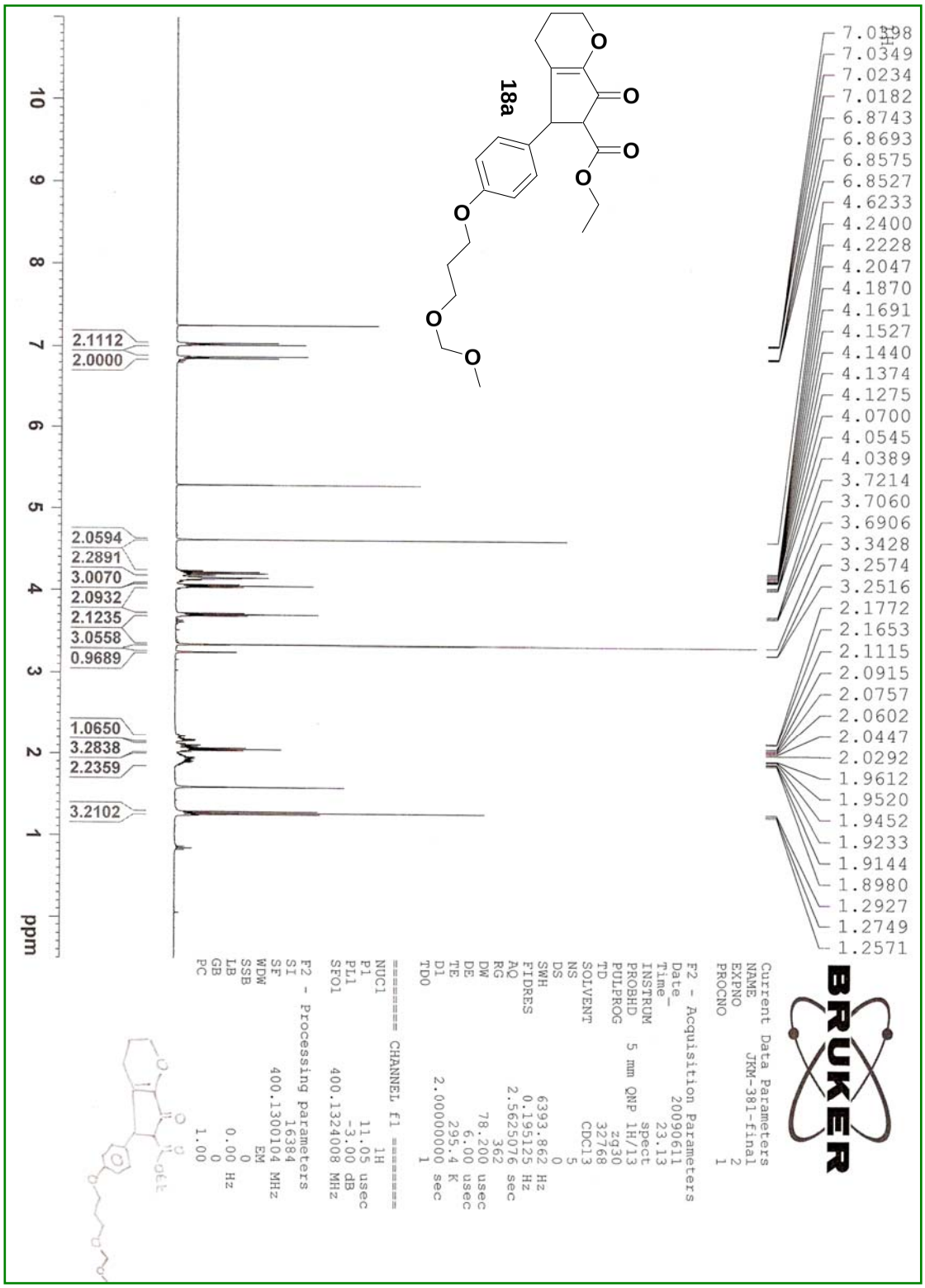

16a
O
O
O
O
Si
2.0000
1.9569
2.1189
2.7634
0.9251
1.0789
1.0275
2.1530
3.0944
9.2418
5.7190
ppm
6.9773
6.9563
6.7924
6.7714
5.2870
4.2346
4.2174
4.1997
4.1817
4.1704
4.1586
4.1407
4.1353
4.1185
3.2580
3.2536
2.2186
2.2025
2.1864
2.1710
2.1575
2.1150
2.1001
2.0850
2.0524
2.0370
1.9882
1.9680
1.9527
1.9432
1.9369
1.9278
1.9216
1.9143
1.9052
1.8984
1.8892
1.8704
1.8535
1.2889
1.2709
1.2531
0.9626
0.1813
Current Data Parameters
NAME JKM-352-TBS
EXPNO 1
PROCNO 6
F2 - Acquisition Parameters
Date_ 20090504
Time_ 18.12
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 3
DS 0
SWH 6393.862 Hz
FIDRES 0.195125 Hz
AQ 2.5625076 sec
RG 71.8
DW 78.200 usec
DE 6.00 usec
TE 289.5 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.05 usec
PL1 -3.00 dB
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300091 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

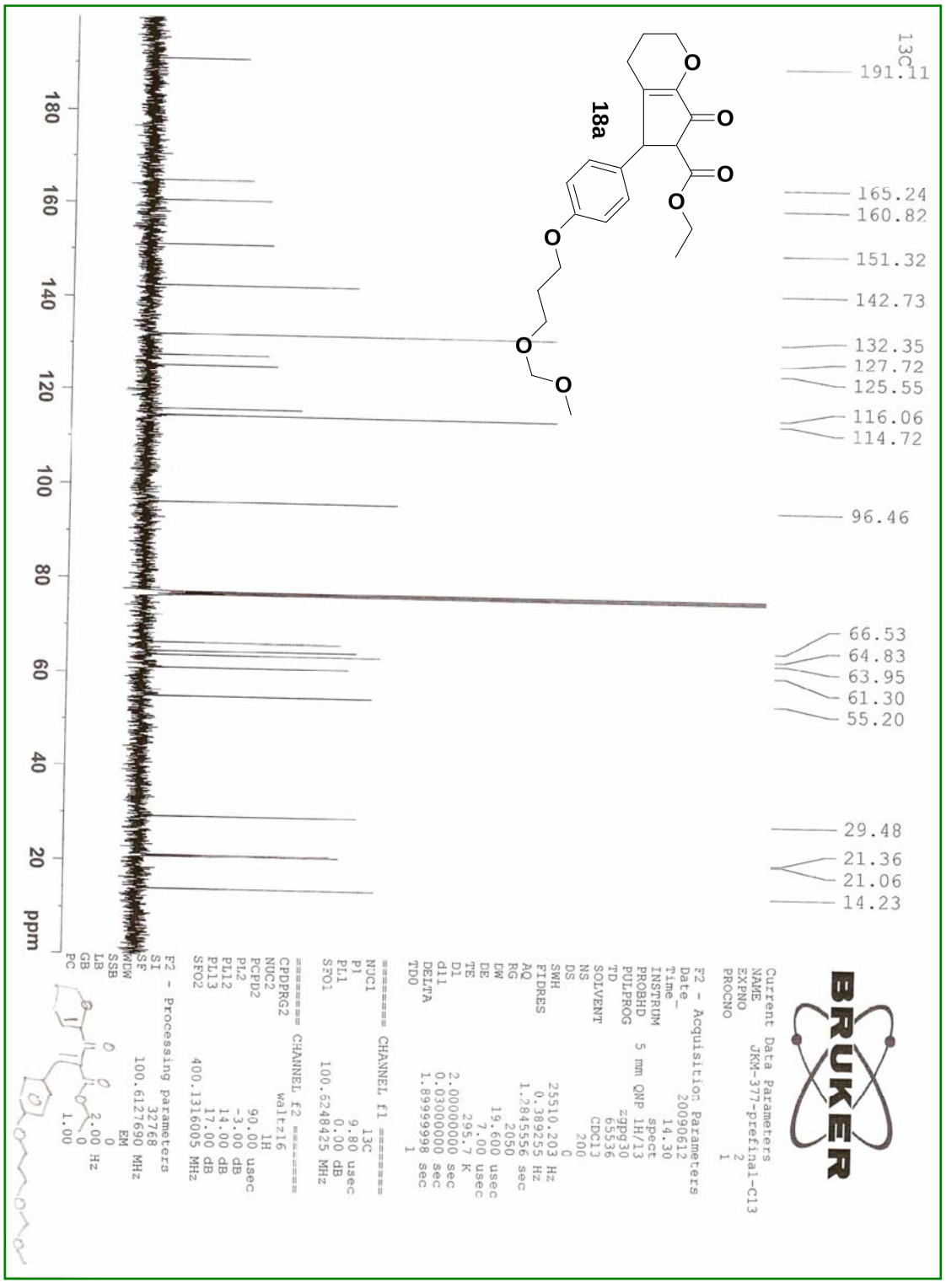

16a
O
O
O
O
O
Si
%T
cm-1
4000.0
3000
2000
1500
1000
400.0
71.2
70
68
66
64
62
60
58
56
54
52
50
48
46
44
42
40
38
36
34
32
30
28.1
2955.89
2931.24
2858.57
1737.15
1714.68
1650.70
1607.87
1509.00
1471.87
1400.05
1367.62
1262.58
1168.91
1123.15
1050.70
1012.57
914.43
841.25
804.37
781.74
702.17

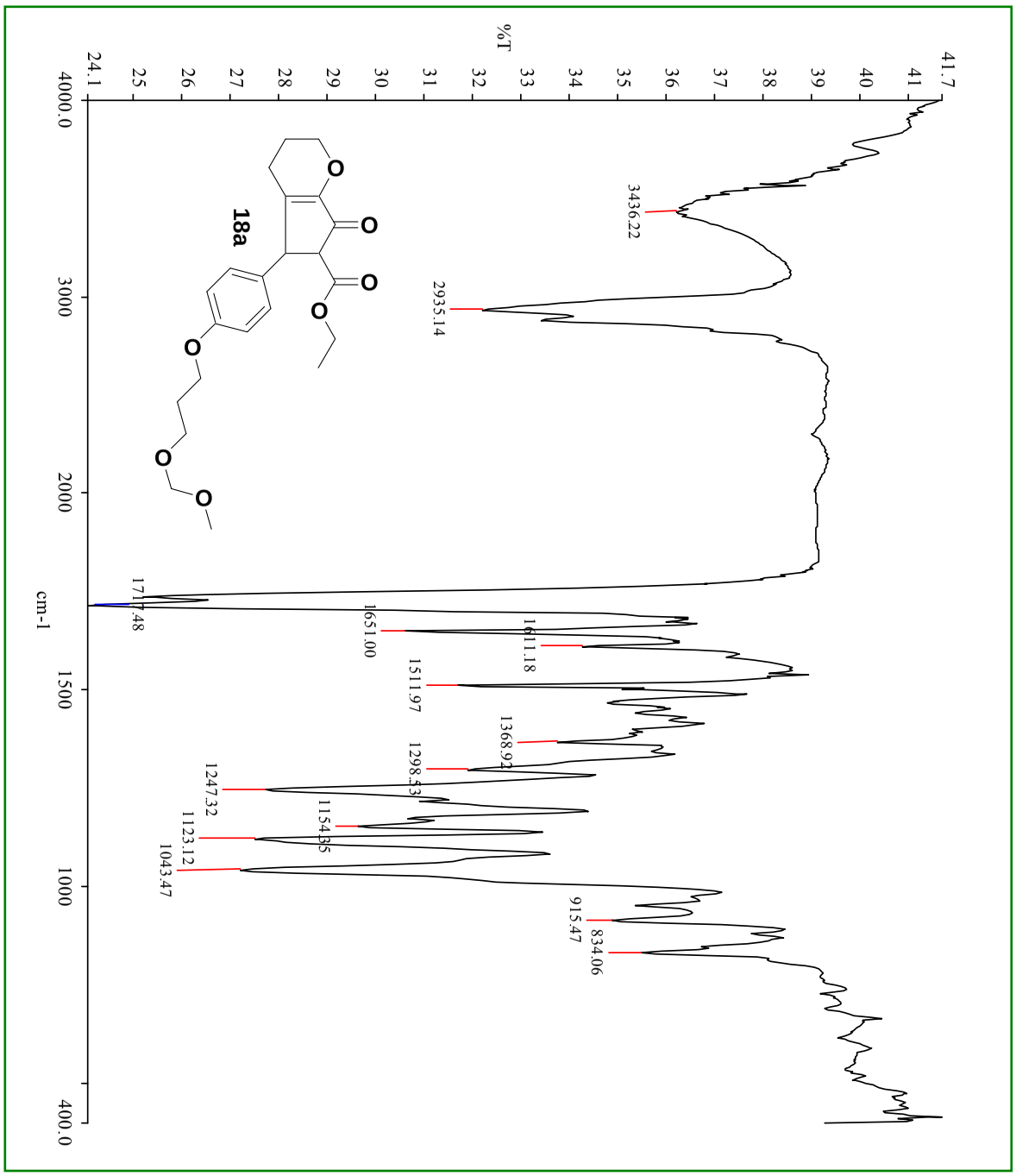

17a
7.0455
7.0309
7.0187
7.0140
6.8732
6.8682
6.8562
6.8515
4.5932
4.5867
4.5757
4.2358
4.2183
4.2007
4.1828
4.1492
4.0766
4.0711
4.0608
4.0556
4.0450
4.0400
3.9228
3.8982
3.8366
3.5778
3.5531
3.2558
3.2501
2.0928
2.0769
2.0613
2.0457
1.6959
1.5793
1.5647
1.5593
1.5434
1.5345
1.5242
1.5019
1.2891
1.2714
1.2533
2.0000
1.9419
1.0560
2.1897
2.9419
2.0225
1.0809
1.0893
1.0821
1.0896
0.9210
1.0509
3.0233
2.1788
1.0800
1.2783
4.2882
3.1119
ppm
BRUKER
Current Data Parameters
NAME JKM-386-THP-final
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090711
Time_ 14.16
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG 2930
TD 32768
SOLVENT CDCl3
NS 5
DS 0
SWH 6393.862 Hz
FIDRES 0.195125 Hz
AQ 2.5625076 sec
RG 114
DW 78.200 usec
DE 6.00 usec
TE 293.8 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.05 usec
PL1 -3.00 dB
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300086 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

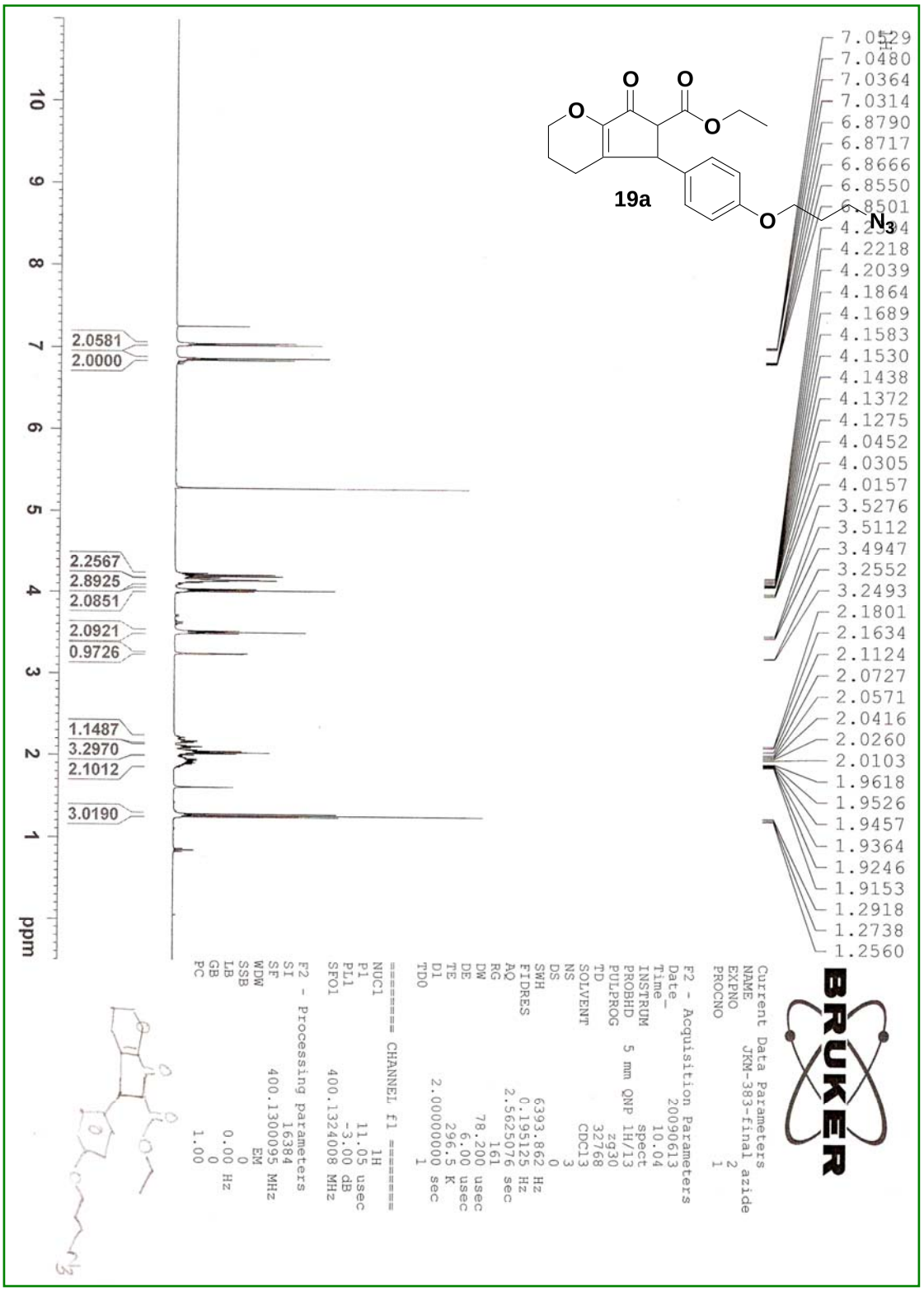

17a
193.30
168.49
158.51
149.67
147.74
131.66
128.37
115.12
98.99
67.07
65.02
63.97
62.39
61.82
59.59
47.03
30.69
29.65
25.44
22.21
21.32
19.63
14.19
180
160
140
120
100
80
60
40
20
ppm
BRUKER

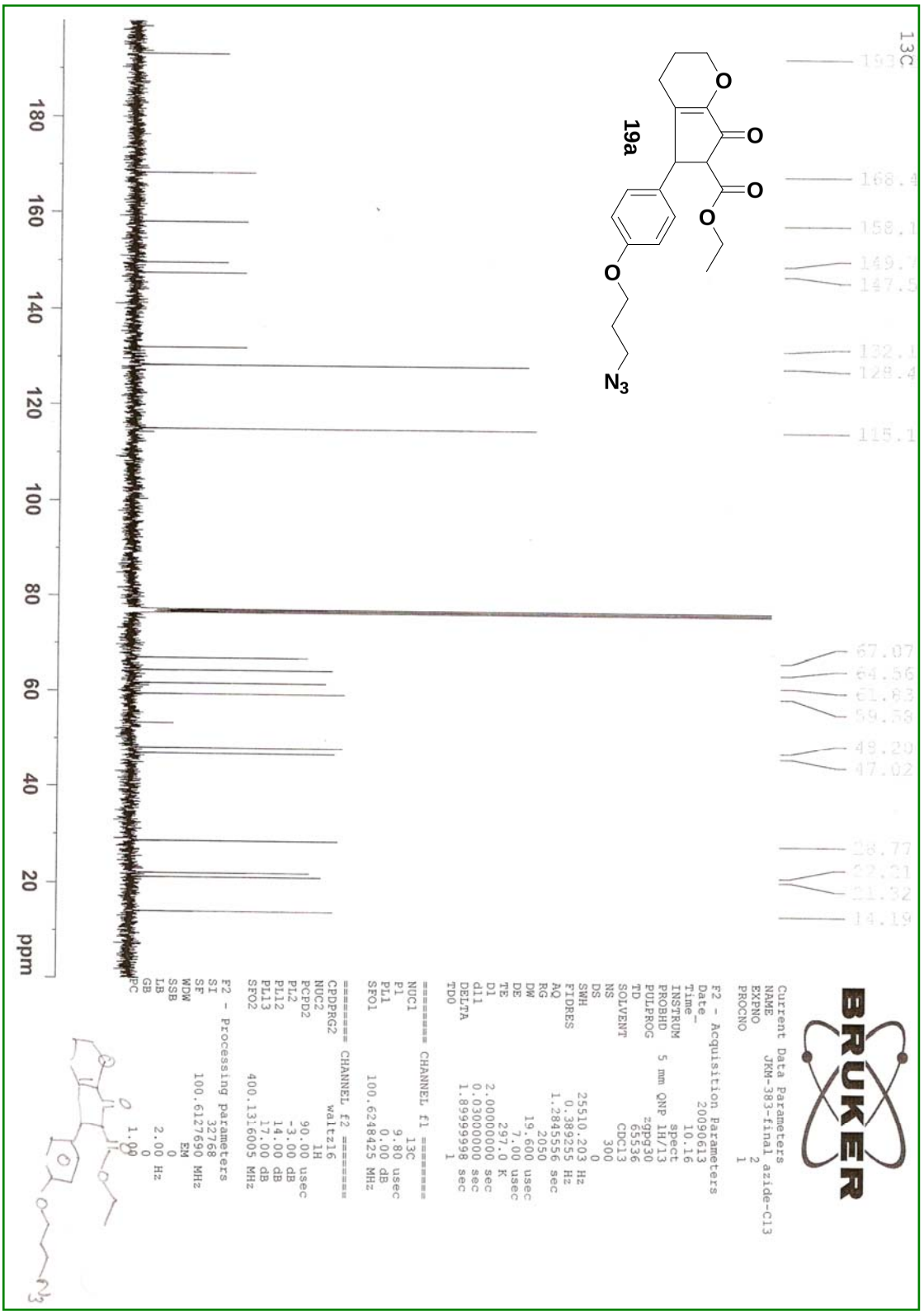

17a
%T
cm-1
70.6
68
66
64
62
60
58
56
54
52
50
48
46
44
42
39.7
4000.0
3000
2000
1500
1000
400.0
2935.52
1717.48
1649.22
1610.52
1511.59
1467.54
1368.36
1298.25
1247.92
1175.97
1123.04
1066.24
1034.53
909.69
833.18

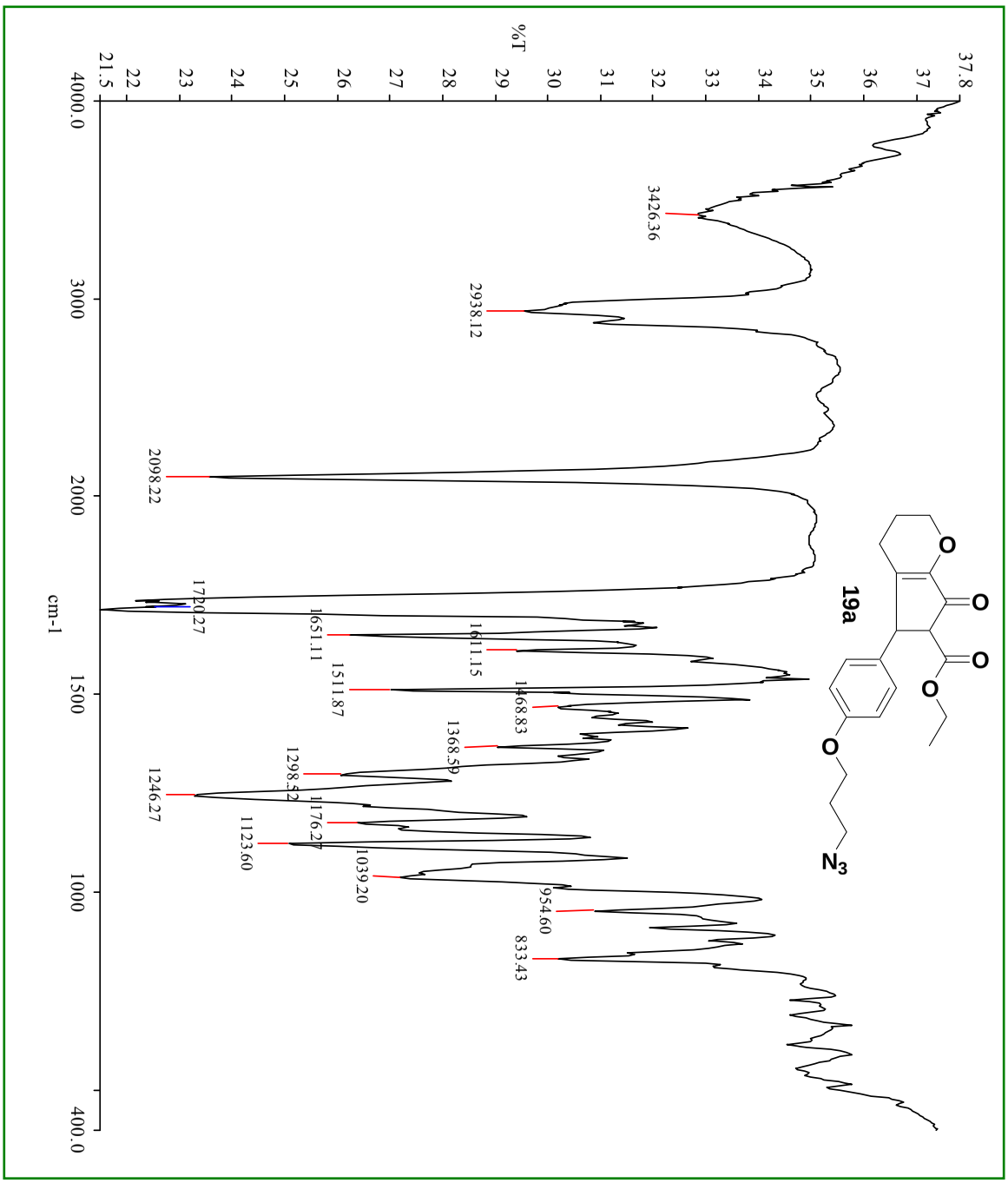

18a
2.1112
2.0000
2.0594
2.2891
3.0070
2.0932
2.1235
3.0558
0.9689
1.0650
3.2838
2.2359
3.2102
ppm
7.0298
7.0349
7.0234
7.0182
6.8743
6.8693
6.8575
6.8527
4.6233
4.2400
4.2228
4.2047
4.1870
4.1691
4.1527
4.1440
4.1374
4.1275
4.0700
4.0545
4.0389
3.7214
3.7060
3.6906
3.3428
3.2574
3.2516
2.1772
2.1653
2.1115
2.0915
2.0757
2.0602
2.0447
2.0292
1.9612
1.9520
1.9452
1.9233
1.9144
1.8980
1.2927
1.2749
1.2571
BRUKER
Current Data Parameters
NAME JKM-381-final
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090611
Time_ 23.13
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 5
DS 0
SWH 6393.862 Hz
FIDRES 0.195125 Hz
AQ 2.5625076 sec
RG 362
DW 78.200 usec
DE 6.00 usec
TE 295.4 K
D1 2.00000000 sec
TD0 1
========== CHANNEL f1 ==========
NUC1 1H
P1 11.05 usec
PL1 -3.00 dB
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300104 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00


18a
13C
191.11
165.24
160.82
151.32
142.73
132.35
127.72
125.55
116.06
114.72
96.46
66.53
64.83
63.95
61.30
55.20
29.48
21.36
21.06
14.23
ppm
BRUKER


%T
cm-1
3436.22
2935.14
1717.48
1651.00
1611.18
1511.97
1368.92
1298.43
1247.32
1154.35
1123.12
1043.47
915.47
834.06
18a


19a
N3
7.0529
7.0480
7.0364
7.0314
6.8790
6.8717
6.8666
6.8550
6.8501
4.2218
4.2039
4.1864
4.1689
4.1583
4.1530
4.1438
4.1372
4.1275
4.0452
4.0305
4.0157
3.5276
3.5112
3.4947
3.2552
3.2493
2.1801
2.1634
2.1124
2.0727
2.0571
2.0416
2.0260
2.0103
1.9618
1.9526
1.9457
1.9364
1.9246
1.9153
1.2918
1.2738
1.2560
2.0581
2.0000
2.2567
2.8925
2.0851
2.0921
0.9726
1.1487
3.2970
2.1012
3.0190
ppm
BRUKER
Current Data Parameters
NAME JKM-383-final azide
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090613
Time_ 10.04
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 3
DS 0
SWH 6393.862 Hz
FIDRES 0.195125 Hz
AQ 2.5625076 sec
RG 161
DW 78.200 usec
DE 6.00 usec
TE 296.5 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.05 usec
PL1 -3.00 dB
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00


19a
13C
ppm
BRUKER


%T
cm-1
37.8
36
35
34
33
32
31
30
29
28
27
26
25
24
23
22
21.5
4000.0
3000
2000
1500
1000
400.0
3426.36
2938.12
2098.22
1720.27
1651.11
1611.15
1511.87
1468.83
1368.59
1298.42
1246.27
1176.27
1123.60
1039.20
954.60
833.43
19a
O
O
O
O
O
N3