Supporting Information

3-Methoxalylchromones – Versatile Reagents for the Regioselective Synthesis of Functionalized Benzophenones, potent UV-filters.

Viktor O. Iaroshenko,[a,b]* Alina Bunescu,[a,c] Anke Spannenberg,[c] Linda Supe,[a] Maria Milyutina[a] Peter Langer[a,c]*

[a]*Institut für Chemie, Universität Rostock,, Albert-Einstein-Str. 3a, 18059 Rostock, Germany. Fax: 00 49 381 49864112; Tel: 0049 381 4986410; E-mail: viktor.iaroshenko@uni-rostock.de, iva108@googlemail.com,*
[b]*National Taras Shevchenko University, 62 Volodymyrska st., Kyiv-33, 01033, Ukraine.E-mail: iva108@googlemail.com.*
[c] *Leibniz-Institut für Katalyse e. V. an der Universität Rostock,, Albert-Einstein-Str. 29a, 18059 Rostock, Germany.*

Table of Contents

(A) Experimental Section

All solvents were purified and dried by standard methods. NMR spectra were recorded on a Brucker AV 300 and Brucker AV400. The following abbreviations were used to designate chemical shift multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet. IR spectra were recorded on a Perkin Elmer FT IR 1600 spectrometer (ATR). Mass spectra were obtained on a "Hewlett-Packard" HP GC / MS 5890 / 5972 instrument (EI, 70 eV) by GC inlet or on a MX-1321 instrument (EI, 70 eV) by direct inlet. Column chromatography was performed on silica gel (63 – 200 mesh, Merck). Silica gel Merck $60F_{254}$ plates were used for TLC. Satisfactory microanalysis obtained C ± 0.33; H ± 0.45; N ± 0.25. Chemical yields refer to pure isolated substances.

Table 1. 1,3-bis-silyl enol ethers **5**.

5	**R_3**	**R_4**
a	Me	H
b	Et	H
c	Bn	H
d	*i*Pr	H
e	*n*Bu	H
f	*i*Bu	H
g	*i*Pent	H
h	*n*Oct	H
i	Me	Me
j	Me	Et
k	Me	*n*Non
l	Me	*n*Tetradec
m	Me	*n*Hexadec
n	Me	$(CH_2)_2Ph$
o	Me	$(CH_2)_3Cl$
p	Me	$(CH_2)_4Cl$

*General procedure for the synthesis of **6** and **7***

Me_3SiOTf (2.0 equiv) was added to chromone **3** (1.0 equiv) at 20 °C. After stirring for 1 h, CH_2Cl_2 (4 mL) the 1,3-bissilylenol ether **5** (2.0 equiv) were added at 0 °C. The mixture was stirred for 12 h at 0-20 °C and was then poured into an aqueous solution of hydrochloric acid (10%). The organic layer was separated and the aqueous layer was extracted with CH_2Cl_2 (2 x 10 mL). The combined organic layers were dried (Na_2SO_4), filtered, and the filtrate was concentrated in vacuo. The residue was purified by chromatography.

*General procedure for the synthesis of **8***

Me_3SiOTf (2.0 equiv) was added to chromone **3** (1.0 equiv) at 20 °C. After stirring for 1 h, CH_2Cl_2 (4 mL) the 1,3-bissilylenol ether **5** (2.0 equiv) were added at 0 °C. The mixture was stirred for 12 h at 0-20 °C and was then poured into an aqueous solution of hydrochloric acid (10%). The organic layer was separated and the aqueous layer was extracted with CH_2Cl_2 (2 x 10 mL). The combined organic layers were dried (Na_2SO_4), filtered, and the filtrate was concentrated in vacuo. To the mixture was added p-TsOH (3 mol%) and was heated at 80-90°C in EtOH (4-8 mL/1 mmol of **3**) during 5-10 h. The solvent was removed in vacuo and the product was purified by chromatography.

*General procedure for the synthesis of **9***

To a CH_2Cl_2 solution (10 mL/mmol **8**) of **8** (1.0 equiv) was added pyridine (4.0 equiv) at –78°C under argon atmosphere. After stirring for 10 min, Tf_2O (2.4 equiv) was added at –78°C. The mixture was allowed to warm to 0°C and stirred for 4 h. The reaction mixture was extracted with water. The organic layer was separated, dried (Na_2SO_4), filtered and the filtrate and was concentrated in vacuo. Products were isolated by column chromatography.

General Procedure for double Suzuki reactions

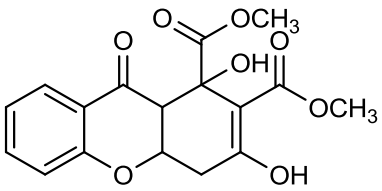
A 1,4-dioxane (5 mL/mmol **9a**) solution of the arylboronic acid (2.0 equiv), K_3PO_4 (3.0 equiv), 6mol% $Pd(PPh_3)_4$, and **9a** (1.0 equiv) was stirred at 90°C for 4 h under argon atmosphere. After cooling to 20 °C, the reaction mixture was poured into water. The organic and the aqueous layer were separated, and the latter was extracted with CH_2Cl_2. The combined organic layers were dried (Na_2SO_4), filtered and the filtrate was concentrated in vacuo. The residue was purified by column chromatography.

General Procedure for triple Suzuki reactions

A 1,4-dioxane (5 mL/mmol **9b**) solution of the arylboronic acid (4.0 equiv), KF (4.5 equiv), 6mol% $Pd(PPh_3)_4$, and **9b** (1.0 equiv) was stirred at 90°C for 4 h under argon atmosphere. After cooling to 20 °C, the reaction mixture was poured into water. The organic and the aqueous layer were separated, and the latter was extracted with CH_2Cl_2. The combined organic layers were dried (Na_2SO_4), filtered and the filtrate was concentrated in vacuo. The residue was purified by column chromatography.

(B) Spectral data

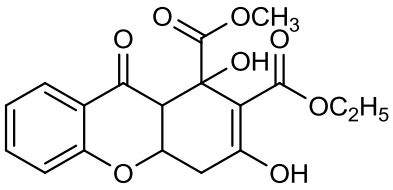
Dimethyl 1,3-dihydroxy-9-oxo-4,4a,9,9a-tetrahydro-1H-xanthene-1,2-dicarboxylate (6a).



Starting with 3-methoxalylchromone (**3a**) (0.232 g, 1.0 mmol) and 1-methoxy-1,3-bis(trimethylsilyloxy)-1,3-butadiene (**5a**) (0.520 g, 2.0 mmol), Me_3SiOTf (0.36 mL, 2.0 mmol) in CH_2Cl_2 (4 mL), the product **6a** was isolated as a white solid (0.183 g, 52%); mp = 170-171 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 2.90-3.16 (m, 2H, CH_2), 3.24 (d, ^3J = 13.5 Hz, 1H, CH), 3.76 (s, 3H, OCH_3), 3.87 (s, 3H, OCH_3), 4.12 (bs, 1H, OH), 4.90-5.00 (m, 1H, CH), 6.96-7.06 (m, 2H, Ar), 7.46-7.52 (m, 1H, Ar), 7.84-7.87 (m, 1H, Ar), 13.00 (s, 1H, OH). ^{13}C NMR (75 MHz, $CDCl_3$): δ = 35.9 (CH_2), 52.0, 53.4, 54.9 (CH/OCH_3), 71.8 (CH), 73.0 (C), 101.9 (C), 117.7 (Ar), 121.2 (C), 122.0, 127.3, 136.5 (Ar), 160.4, 171.0, 172.7, 174.0, 189.8

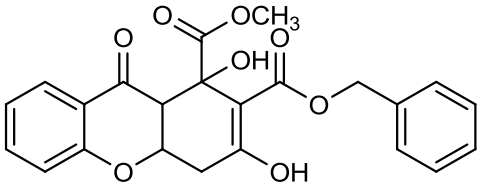
(C). IR (ATR, cm^{-1}): $\tilde{\nu}$ = 3459 (m), 2950 (w), 2903 (w), 1746 (s), 1726 (s), 1683 (s), 1655 (m), 1607 (s), 1466 (s), 1219 (s), 1083 (s), 854 (s), 764 (s), 466 (s). HRMS (ESI): calcd. for $C_{17}H_{17}O_8$ ($[M+H]^+$) 349.0918, found 349.0921; calcd. for $C_{17}H_{16}NaO_8$ ($[M+Na]^+$) 371.0737, found 371.0743. Anal. calcd. for $C_{17}H_{16}O_8$ (348.30): C, 58.62; H, 4.63. Found: C, 58.64; H, 4.69.

2-Ethyl 1-methyl 1,3-dihydroxy-9-oxo-4,4a,9,9a-tetrahydro-1H-xanthene-1,2-dicarboxylate (6b).



Starting with 3-methoxalylchromone (**3a**) (0.232 g, 1.0 mmol) and 1-ethoxy-1,3-bis(trimethylsilyloxy)-1,3-butadiene (**5b**) (0.549 g, 2.0 mmol), Me_3SiOTf (0.36 mL, 2.0 mmol) in CH_2Cl_2 (4 mL), the product **6b** was isolated as a white solid (0.173 g, 47%); mp = 159-160 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.28 (t, ^3J = 7.1 Hz, 3H, CH_3CH_2), 2.89-3.15 (m, 2H, CH_2), 3.24 (d, ^3J = 13.5 Hz, 1H, CH), 3.87 (s, 3H, OCH_3), 4.13 (bs, 1H, OH), 4.15-4.35 (m, 2H, CH_3CH_2), 4.90-5.00 (m, 1H, CH), 6.95-7.06 (m, 2H, Ar), 7.46-7.52 (m, 1H, Ar), 7.84-7.87 (m, 1H, Ar), 13.11 (s, 1H, OH). ^{13}C NMR (75 MHz, $CDCl_3$): δ = 13.9 (CH_3), 35.9 (CH_2), 53.4, 55.1 (CH/OCH_3), 61.3 (CH_2), 71.8 (CH), 73.0 (C), 101.9 (C), 117.7 (Ar), 121.2 (C), 122.0, 127.3, 136.4 (Ar), 160.4, 170.6, 172.5, 174.0, 189.7 (C). IR (ATR, cm^{-1}): $\tilde{\nu}$ = 3463 (m), 2975 (w), 1743 (s), 1693 (m), 1637 (m), 1605 (s), 1578 (m), 1230 (s), 1095 (s), 760 (s), 577 (s). MS (EI, 70 eV): *m/z* (%): 362 (M^+, 3), 303 (35), 257 (100), 160 (16), 121 (66). HRMS (ESI): calcd. for C18H18NaO8 ($[M+Na]^+$) 385.0894, found 385.0901. Anal. calcd. for $C_{18}H_{18}O_8$ (362.33): C, 59.64; H, 5.01. Found: C, 59.70; H, 5.01.

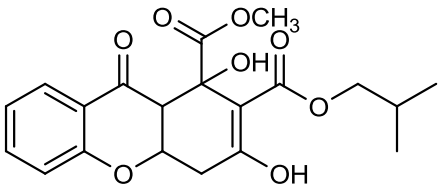
2-Benzyl 1-methyl 1,3-dihydroxy-9-oxo-4,4a,9,9a-tetrahydro-1H-xanthene-1,2-dicarboxylate (6c).



Starting with 3-methoxalylchromone (**3a**) (0.232 g, 1.0 mmol) and 1-benzyloxy-1,3-bis(trimethylsilyloxy)-1,3-butadiene (**5c**) (0.673 g, 2.0 mmol), Me_3SiOTf (0.36 mL, 2.0 mmol) in CH_2Cl_2 (4 mL), the product **6c** was isolated as a slight yellow solid (0.174 g, 41%); mp = 166-168 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 2.90-3.16 (m, 2H, CH_2), 3.23 (d, ^3J = 13.5 Hz, 1H, CH), 3.45 (s, 3H, OCH_3), 4.11 (bs, 1H, OH), 4.89-4.99 (m, 1H, CH), 5.22 (dd, ^2J = 12.0 Hz, 2H, CH_2Ph), 6.95-7.05 (m, 2H, Ar), 7.33-7.39 (m, 5H, Ph), 7.46-7.51 (m, 1H, Ar), 7.82-7.86 (m, 1H, Ar), 13.07 (s, 1H, OH). ^{13}C NMR (75 MHz, $CDCl_3$): δ = 35.9 (CH_2), 52.9, 55.1 (CH/OCH_3), 66.9 (CH_2), 71.8 (CH), 73.1 (C), 101.8 (C), 117.7 (Ar), 121.2 (C), 121.9, 127.3 (Ar), 128.5, 128.6, 128.7, 134.7 (Ph), 136.4 (Ar),

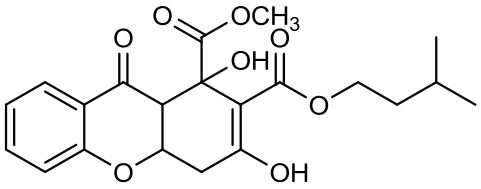
160.3, 170.5, 172.9, 173.7, 189.7 (C). IR (ATR, cm^{-1}): $\tilde{\nu}$ = 3462 (m), 2953 (w), 1751 (s), 1688 (m), 1637 (m), 1603 (s), 1577 (m), 1285 (s), 1229 (s), 757 (s), 697 (s), 586 (s). MS (EI, 70 eV): *m/z* (%): 424 (M^+, 3), 365 (43), 257 (38), 121 (23), 91 (100). HRMS (EI, 70 eV): calcd. for $C_{23}H_{20}O_8$ (M^+) 424.11527, found 424.11508. Anal. calcd. for $C_{23}H_{20}O_8$ (424.40): C, 65.09; H, 4.75. Found: C, 65.13; H, 4.66.

2-Isobutyl 1-methyl 1,3-dihydroxy-9-oxo-4,4a,9,9a-tetrahydro-1H-xanthene-1,2-dicarboxylate (6d).



Starting with 3-methoxalylchromone (**3a**) (0.232 g, 1.0 mmol) and 1-isobutyloxy-1,3-bis(trimethylsilyloxy)-1,3-butadiene (**5f**) (0.605 g, 2.0 mmol), Me_3SiOTf (0.36 mL, 2.0 mmol) in CH_2Cl_2 (4 mL), the product **6d** was isolated as a slight yellow solid (0.167 g, 43%); mp = 152-153 °C. ^1H NMR (300 MHz, $CDCl_3$): δ = 0.92-0.97 (m, 6H, $(CH_3)_2$), 1.88-2.00 (m, 1H, C*H*$(CH_3)_2$), 2.89-3.15 (m, 2H, CH_2), 3.24 (d, ^3J = 13.2 Hz, 1H, CH), 3.86 (s, 3H, OCH_3), 3.92-4.01 (m, 2H, C*H*$_2$CH$(CH_3)_2$), 4.15 (bs, 1H, OH), 4.89-4.99 (m, 1H, CH), 6.95-7.05 (m, 2H, Ar), 7.46-7.52 (m, 1H, Ar), 7.84-7.87 (m, 1H, Ar), 13.18 (s, 1H, OH). ^{13}C NMR (75 MHz, $CDCl_3$): δ = 18.9, 19.1 (CH_3), 27.5 (CH), 36.0 (CH_2), 53.4, 55.1 (CH/OCH_3), 71.7 (CH_2), 71.8 (CH), 73.0 (C), 101.8 (C), 117.7 (Ar), 121.3 (C), 122.0, 127.3, 136.4 (Ar), 160.4, 170.9, 172.6, 173.8, 189.7 (C). IR (ATR, cm^{-1}): $\tilde{\nu}$ = 3476 (m), 2961 (w), 1749 (s), 1690 (m), 1633 (m), 1605 (s), 1460 (m), 1294 (s), 1230 (s), 1076 (s), 764 (s). MS (EI, 70 eV): *m/z* (%): 390 (M^+, 2), 331 (35), 257 (100), 121 (32). HRMS (EI, 70 eV): calcd. for $C_{20}H_{22}O_8$ (M^+) 390.13092, found 390.13090. Anal. calcd. for $C_{20}H_{22}O_8$ (390.38): C, 61.53; H, 5.68. Found: C, 61.44; H, 5.62.

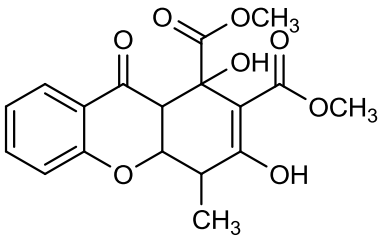
2-Isopentyl 1-methyl 1,3-dihydroxy-9-oxo-4,4a,9,9a-tetrahydro-1H-xanthene-1,2-dicarboxylate (6e).



Starting with 3-methoxalylchromone (**3a**) (0.232 g, 1.0 mmol) and 1-isopentyloxy-1,3-bis(trimethylsilyloxy)-1,3-butadiene (**5g**) (0.633 g, 2.0 mmol), Me_3SiOTf (0.36 mL, 2.0 mmol) in CH_2Cl_2 (4 mL), the product **6e** was isolated as a white solid (0.210 g, 52%); mp = 137-138 °C. ^1H NMR (300 MHz, $CDCl_3$): δ = 0.92 (t, ^3J = 6.5 Hz, 6H, $(CH_3)_2$), 1.46-1.59 (m, 2H, CH_2CH_2CH), 1.61-1.76 (m, 1H, C*H*$(CH_3)_2$), 2.89-3.14 (m, 2H, (CH_2)), 3.22 (d, ^3J = 13.4 Hz, 1H, CH), 3.86 (s, 3H, OCH_3), 4.12 (bs, 1H, OH), 4.20 (t, ^3J = 6.9 Hz, 2H, OC*H*$_2$), 4.89-4.99 (m, 1H, CH), 6.95-7.05 (m, 2H, Ar), 7.46-7.52 (m, 1H, Ar), 7.84-7.87 (m, 1H, Ar), 13.13 (s, 1H, OH). ^{13}C NMR (75 MHz, $CDCl_3$): δ = 22.2, 22.4 (CH_3), 24.7 (CH), 35.9, 36.9 (CH_2), 53.3, 55.0 (CH/OCH_3), 64.1 (CH_2), 71.8 (CH), 73.0 (C),

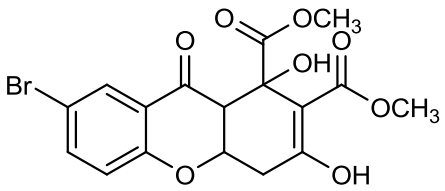
101.9 (C), 117.7 (Ar), 121.3 (C), 122.0, 127.3, 136.4 (Ar), 160.4, 170.8, 172.5, 173.9, 189.7 (C). IR (ATR, cm^{-1}): $\tilde{\nu}$ = 3476 (m), 2958 (m), 1741 (s), 1687 (s), 1639 (m), 1603 (s), 1463 (m), 1241 (s), 1077 (s), 859 (s), 762 (s). MS (EI, 70 eV): *m/z* (%): 404 (M^+, 2), 345 (43), 257 (100), 121 (33). HRMS (EI, 70 eV): calcd. for $C_{21}H_{24}O_8$ (M^+) 404.14657, found 404.14628. Anal. calcd. for $C_{10}H_{24}O_8$ (390.38): C, 62.37; H, 5.98. Found: C, 62.27; H, 5.80.

Dimethyl 1,3-dihydroxy-4-methyl-9-oxo-4,4a,9,9a-tetrahydro-1H-xanthene-1,2-dicarboxylate (6f).



Starting with 3-methoxalylchromone (**3a**) (0.232 g, 1.0 mmol) and 1-methoxy-1,3-bis(trimethylsilyloxy)-1,3-pentadiene (**5i**) (0.549 g, 2.0 mmol), Me_3SiOTf (0.36 mL, 2.0 mmol) in CH_2Cl_2 (4 mL), the product **6f** was isolated as a white solid (0.152 g, 42%); mp = 156-157 °C. ^1H NMR (400 MHz, $CDCl_3$): δ = 1.46 (d, ^3J = 7.2 Hz, 3H, CH_3), 3.12-3.18 (m, 1H, CH), 3.41 (d, ^3J = 14.0 Hz, 1H, CH), 3.75 (s, 3H, OCH_3), 3.87 (s, 3H, OCH_3), 4.13 (bs, 1H, OH), 5.00-5.05 (m, 1H, CH), 6.96-7.03 (m, 2H, Ar), 7.46-7.50 (m, 1H, Ar), 7.83-7.86 (m, 1H, Ar), 13.06 (s, 1H, OH). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 12.2 (CH_3), 38.2, 49.8 (CH), 52.0, 53.4 (OCH_3), 73.0 (C), 73.5 (CH), 100.6 (C), 117.8 (Ar), 121.0 (C), 121.8, 127.2, 136.4 (Ar), 160.5, 171.4, 174.1, 177.1, 190.3 (C). IR (ATR, cm^{-1}): $\tilde{\nu}$ = 3475 (m), 2947 (w), 1476 (s), 1680 (s), 1657 (m), 1607 (s), 1467 (s), 1218 (s), 1101 (s), 1044 (s), 780 (s). MS (EI, 70 eV): *m/z* (%): 362 (M^+, 1), 303 (34), 271 (97), 174 (32), 151 (22), 121 (100). HRMS (EI, 70 eV): calcd. for $C_{18}H_{18}O_8$ (M^+) 362.09962, found 362.09931. Anal. calcd. for $C_{18}H_{18}O_8$ (362.33): C, 59.67; H, 5.01. Found: C, 59.50; H, 4.97.

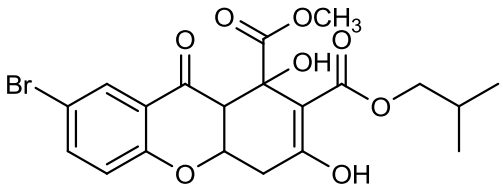
Dimethyl 7-bromo-1,3-dihydroxy-9-oxo-4,4a,9,9a-tetrahydro-1H-xanthene-1,2-dicarboxylate (6g).



Starting with 6-bromo-3-methoxalylchromone (**3a**) (0.309 g, 1.0 mmol) and 1-methoxy-1,3-bis(trimethylsilyloxy)-1,3-butadiene (**5a**) (0.520 g, 2.0 mmol), Me_3SiOTf (0.36 mL, 2.0 mmol) in CH_2Cl_2 (4 mL), the product **6g** was isolated as a slight orange solid (0.220 g, 51%); mp = 160-162 °C. ^1H NMR (300 MHz, $CDCl_3$): δ = 2.89-3.15 (m, 2H, CH_2), 3.22 (d, ^3J = 13.4 Hz, 1H, CH), 3.76 (s, 3H, OCH_3), 3.87 (s, 3H, OCH_3), 4.11 (bs, 1H, OH), 4.88-4.98 (m, 1H, CH), 6.88 (d, ^3J = 8.7 Hz, 1H, Ar), 7.54-7.58 (dd, ^3J = 8.8 Hz, ^4J = 2.5 Hz 1H, Ar), 7.95 (d,

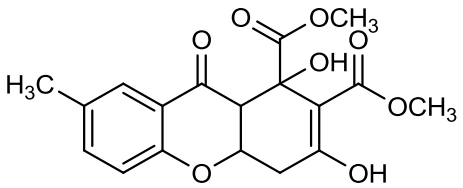
^4J = 2.5 Hz, 1H, Ar), 12.99 (s, 1H, OH). ^{13}C NMR (75 MHz, $CDCl_3$): δ = 35.7 (CH_2), 52.0, 53.5, 54.7 (CH/OCH_3), 72.0 (CH), 72.9 (C), 101.9, 114.7 (C), 119.8 (Ar), 122.4 (C), 129.8, 139.0 (Ar), 159.4, 170.0, 172.4, 173.8, 188.6 (C). IR (ATR, cm^{-1}): $\tilde{\nu}$ = 3461 (m), 2954 (w), 1749 (s), 1695 (m), 1659 (m), 1618 (m), 1597 (m), 1471 (m), 1285 (s), 1218 (s), 1110 (s), 839 (s), 639 (s), 584 (s). HRMS (ESI): calcd. for $C_{17}H_{15}Br^{79}NaO_8$ ([M+Na]$^+$) 448.9842, found 448.9840; calcd. for $C_{17}H_{15}Br^{81}NaO_8$ ([M+Na]$^+$) 450.9824, found 450.9822. Anal. calcd. for $C_{17}H_{15}BrO_8$ (427.20): C, 47.80; H, 3.54. Found: C, 48.05; H, 3.56.

2-Isobutyl 1-methyl 7-bromo-1,3-dihydroxy-9-oxo-4,4a,9,9a-tetrahydro-1H-xanthene-1,2-dicarboxylate (6h).



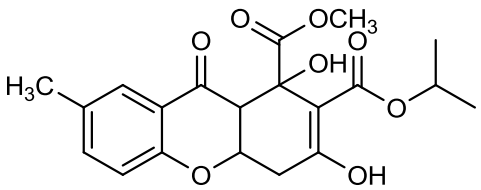
Starting with 6-bromo-3-methoxalylchromone (**3a**) (0.309 g, 1.0 mmol) and 1-isobuoxy-1,3-bis(trimethylsilyloxy)-1,3-butadiene (**5f**) (0.605 g, 2.0 mmol), Me_3SiOTf (0.36 mL, 2.0 mmol) in CH_2Cl_2 (4 mL), the product **6h** was isolated as a white solid (0.375 g, 80%); mp = 129-131 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.92-0.97 (m, 6H, $(CH_3)_2$), 1.88-1.04 (m, 1H, CH), 2.89-3.14 (m, 2H, CH_2), 3.22 (d, ^3J = 13.4 Hz, 1H, CH), 3.86 (s, 3H, OCH_3), 3.88-4.01 (m, 2H, CH_2), 4.14 (bs, 1H, OH), 4.87-4.97 (m, 1H, CH), 6.88 (d, ^3J = 8.8 Hz, 1H, Ar), 7.54-7.58 (dd, ^3J = 8.7 Hz, ^4J = 2.4 Hz 1H, Ar), 7.95 (d, ^4J = 2.5 Hz, 1H, Ar), 13.18 (s, 1H, OH). ^{13}C NMR (75 MHz, $CDCl_3$): δ = 18.9, 19.1 (CH_3), 27.5 (CH), 35.8 (CH_2), 53.5, 54.8 (CH/OCH_3), 71.8 (CH_2), 72.1 (CH), 72.9 (C), 101.8, 114.7 (C), 119.8 (Ar), 122.5 (C), 129.8, 139.0 (Ar), 159.2, 170.8, 172.3, 173.6, 188.5 (C). IR (ATR, cm^{-1}): $\tilde{\nu}$ = 3430 (w), 2952 (w), 2894 (w), 2731 (w), 1755 (m), 1692 (s), 1639 (m), 1598 (s), 1467 (m), 1409 (s), 1231 (s), 1100 (s), 820 (s), 638 (s), 386 (s). MS (EI, 70 eV): *m/z* (%): 470 (M$^+$, 5), 468 (M$^+$, 5), 411 (34), 409 (34), 364 (26), 362 (26), 337 (99), 335 (100), 201 (32), 199 (33). HRMS (ESI): calcd. for $C_{20}H_{21}Br^{79}NaO_8$ ([M+Na]$^+$) 491.0312, found 491.0322; calcd. for $C_{20}H_{21}Br^{81}NaO_8$ ([M+Na]$^+$) 493.0294, found 493.0307. Anal. calcd. for $C_{20}H_{21}BrO_8$ (468.28): C, 51.19; H, 4.51. Found: C, 51.18; H, 4.67.

Dimethyl 1,3-dihydroxy-7-methyl-9-oxo-4,4a,9,9a-tetrahydro-1H-xanthene-1,2-dicarboxylate (6i).



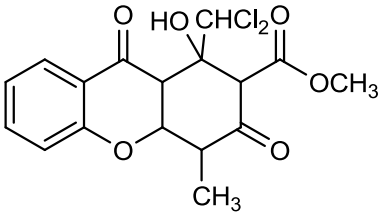
Starting with 6-methyl-3-methoxalylchromone (**3a**) (0.246 g, 1.0 mmol) and 1-methoxy-1,3-bis(trimethylsilyloxy)-1,3-butadiene (**5a**) (0.520 g, 2.0 mmol), Me_3SiOTf (0.36 mL, 2.0 mmol) in CH_2Cl_2 (4 mL), the product **6i** was isolated as a slight orange solid (0.260 g, 71%); mp = 165-167 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 2.29 (s. 3H, CH_3), 2.87-3.24 (m, 2H, CH_2), 3.49-3.54 (m, 1H, CH), 3.78 (s, 3H, OCH_3), 3.89 (s, 3H, OCH_3), 3.92 (bs, 1H, OH), 4.54 (bs, 1H, OH), 4.85-4.95 (m, 1H, CH), 6.88 (d, ^3J = 8.4 Hz, 1H, Ar), 7.29-7.33 (m, 1H, Ar), 7.66-7.67 (m, 1H, Ar). ^{13}C NMR (75 MHz, $CDCl_3$): δ = 20.4 (CH_3), 46.1 (CH_2), 52.7. 53.5, 54.5 (CH/OCH_3), 62.9, 74.6 (CH), 75.5 (C), 117.5 (Ar), 120.5 (C), 127.1 (Ar), 131.8 (C), 137.6 (Ar), 158.8, 167.9, 172.0, 188.3, 197.2 (C). IR (ATR, cm^{-1}): $\tilde{\nu}$ = 3454 (m), 2956 (w), 2923 (w), 1768 (s), 1726 (s), 1682 (s), 1617 (m), 1579 (w), 1488 (s), 1220 (s), 1126 (s), 823 (s), 761 (s), 602 (s), 503 (s). HRMS (ESI): calcd. for $C_{18}H_{19}O_8$ ($[M+H]^+$) 363.1074, found 360.1082; calcd. for $C_{18}H_{18}NaO_8$ ($[M+Na]^+$) 385.0893, found 363.1082. Anal. calcd. for $C_{18}H_{18}O_8$ (362.33): C, 59.67; H, 5.01. Found: C, 59.87; H, 5.12.

2-Isopropyl 1-methyl 1,3-dihydroxy-7-methyl-9-oxo-4,4a,9,9a-tetrahydro-1H-xanthene-1,2-dicarboxylate (6j).



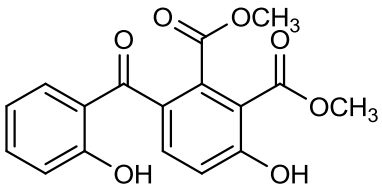
Starting with 6-methyl-3-methoxalylchromone (**3a**) (0.246 g, 1.0 mmol) and 1-isopropoxy-1,3-bis(trimethylsilyloxy)-1,3-butadiene (**5d**) (0.577 g, 2.0 mmol), Me_3SiOTf (0.36 mL, 2.0 mmol) in CH_2Cl_2 (4 mL), the product **6j** was isolated as a slight orange solid (0.176 g, 45%); mp = 128-130 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.24-1.29 (m, 6H, $(CH_3)_2$), 2.28 (s. 3H, CH_3), 2.86-3.12 (m, 2H, CH_2), 3.20 (d, ^3J = 13.4 Hz, 1H, CH), 3.87 (s, 3H, OCH_3), 4.11 (bs, 1H, OH), 4.84-4.94 (m, 1H, CH), 5.06-5.15 (m, 1H, CH), 6.87 (d, ^3J = 8.4 Hz, 1H, Ar), 7.28-7.31 (m, 1H, Ar), 7.64 (d, ^4J = 2.2 Hz, 1H, Ar), 13.18 (s, 1H, OH). ^{13}C NMR (75 MHz, $CDCl_3$): δ = 20.4, 21.4, 21.6 (CH_3), 35.9 (CH_2), 53.2, 55.1 (CH/OCH_3), 69.3, 71.8 (CH), 73.0 102.7 (C), 117.4 (Ar), 120.9 (C), 126.8 (Ar), 131.4 (C), 137.4 (Ar), 158.5, 170.2, 172.4, 174.0, 189.9 (C). IR (ATR, cm^{-1}): $\tilde{\nu}$ = 3493 (w), 2980 (w), 2957 (w), 1734 (m), 1684 (s), 1612 (s), 1487 (m), 1441 (w), 1421 (w), 1217 (s), 1095 (s), 832 (s), 796 (s), 586 (s). MS (EI, 70 eV): *m/z* (%): 390 (M^+, 4), 331 (37), 271 (100), 135 (71). HRMS (EI): calcd. for $C_{20}H_{22}O_8$ $[M]^+$ 390.1309, found 390.1306. Anal. calcd. for $C_{20}H_{22}O_8$ (390.38): C, 61.53; H, 5.68. Found: C, 61.50; H, 5.56.

Methyl 1-(dichloromethyl)-1-hydroxy-4-methyl-3,9-dioxo-2,3,4,4a,9,9a-hexahydro-1H-xanthene-2-carboxylate (7).



Starting with 3-dichloroacetylchromone (**4**) (0.257 g, 1.0 mmol) and 1-methoxy-1,3-bis(trimethylsilyloxy)-1,3-pentadiene (**5i**) (0.549 g, 2.0 mmol), Me_3SiOTf (0.36 mL, 2.0 mmol) in CH_2Cl_2 (4 mL), a mixture of isomers of **7** was isolated as a white solid (0.210 g, 54%); mp = 186-188 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.35 (d, ^3J = 6.3 Hz, 0.3H, CH_3), 1.39 (d, ^3J = 6.3 Hz, 3H, CH_3), 2.96-3.06 (m, 1H, CH), 3.53-3.58 (m, 1H, CH), 3.75 (s, 0.8H, OCH_3), 3.87 (s, 3H, OCH_3), 4.18 (bs, 0.3H, OH), 4.19 (bs, 1H, OH), 4.26-4.40 (m, 0.4H, CH), 4.42-4.50 (m, 0.4H, CH), 5.8 (d, ^4J = 2.0 Hz, 1H, CH), 6.99-7.03 (m, 0.6H, Ar), 7.03-7.11 (m, 2H, Ar), 7.48 (bs, 0.3H, Ar), 7.49-7.56 (m, 1H, Ar), 7.78 (s, 1H, *CH*Cl_2), 7.86-7.89 (m, 0.3H, Ar), 7.92-7.95 (m, 1H, Ar). ^{13}C NMR (75 MHz, $CDCl_3$): δ = 10.2, 10.8 (CH_3), 46.6, 48.3, 50.2, 51.9, 53.1, 53.3 (OCH_3), 56.7, 62.1, 75.9 (CH), 79.8 (C), 79.9 (CH), 81.2 (C), 81.7 (CH), 117.4, 117.6 (Ar), 121.5 (C), 121.9, 122.4, 127.6, 128.0, 136.6 (Ar), 160.0, 170.7, 189.0, 200.4 (C). IR (ATR, cm^{-1}): $\tilde{\nu}$ = 3850 (w), 3732 (w), 3667 (w), 3646 (w), 3626 (w), 3328 (w), 3042 (w), 2942 (w), 2874 (w), 1731 (s), 1704 (m), 1667 (s), 1603 (s), 1581 (m), 1308 (s), 1201 (s), 767 (s), 611 (s). MS (ESI, 70 eV): *m/z* (%): 409.0214 ([M+Na]$^+$). Anal. calcd. for $C_{16}H_{14}Cl_2O_6$ (373.18): C, 51.49; H, 3.78. Found: C, 51.81; H, 4.24.

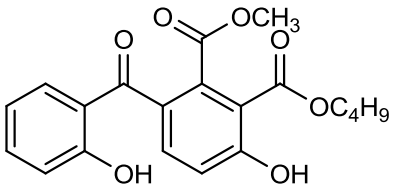
Dimethyl 3-hydroxy-6-(2-hydroxybenzoyl)phthalate (8a).



Starting with 3-methoxalylchromone (**3a**) (0.232 g, 1.0 mmol) and 1-methoxy-1,3-bis(trimethylsilyloxy)-1,3-butadiene (**5a**) (0.520 g, 2.0 mmol), Me_3SiOTf (0.36 mL, 2.0 mmol) in CH_2Cl_2 (4 mL) and then 10 h heating in EtOH (4 mL) with 3 mol% of p-TsOH, the product **8a** was isolated as a yellow solid (0.208 mg, 63%); mp = 153-154 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 3.77 (s, 3H, OCH_3), 3.49 (s, 3H, OCH_3), 6.82-6.88 (m, 1H, Ar), 7.02-7.06 (dd, ^3J = 8.4 Hz, ^4J = 0.9 Hz 1H, Ar), 7.11 (d, ^3J = 8.7 Hz, 1H, Ar), 7.37-7.40 (dd, ^3J = 8.0 Hz, ^4J = 1.6 Hz, 1H, Ar), 7.47-7.53 (m, 1H, Ar), 7.56 (d, ^3J = 8.6 Hz, 1H, Ar), 11.08 (s, 1H, OH), 11.62 (s, 1H, OH). ^{13}C NMR (75 MHz, $CDCl_3$): δ = 52.7, 53.3 (OCH_3), 111.0 (C), 118.4, 118.8 (Ar), 119.0, 128.3 (C), 133.1, 135.3 (Ar), 136.4 (C), 136.8 (Ar), 163.0, 163.1, 167.5, 168.9, 199.6 (C). IR (ATR, cm^{-1}): $\tilde{\nu}$ = 3291 (w), 2961 (w), 1731 (m), 1683 (s), 1626 (s), 1609 (m), 1590 (m), 1442 (s), 1320 (s), 1184(s), 1119 (s), 1012 (s), 759 (s), 719 (s), 638 (s), 530 (s),

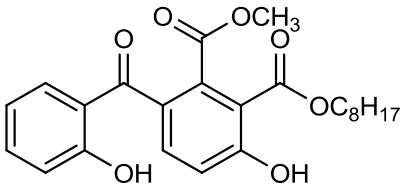
388 (s). GC-MS (EI, 70 eV): *m/z* (%) = 330 (M^+, 7), 298 (37), 239 (100). HRMS (EI, 70 eV): calcd. for $C_{17}H_{14}O_7$ (M^+) 330.07340, found 330.07361. Anal. calcd. for $C_{17}H_{14}O_7$ (330.29): C, 61.82; H, 4.27. Found: C, 61.54; H, 4.28.

1-Butyl 2-methyl 6-hydroxy-3-(2-hydroxybenzoyl)phthalate (8b).



Starting with 3-methoxalylchromone (**3a**) (0.232 g, 1.0 mmol) and 1-butoxy-1,3-bis(trimethylsilyloxy)-1,3-butadiene (**5e**) (0.605 g, 2.0 mmol), Me_3SiOTf (0.36 mL, 2.0 mmol) in CH_2Cl_2 (4 mL) and then 10 h heating in EtOH (4 mL) with 3 mol% of p-TsOH, the product **8b** was isolated as a yellow oil (0.311 mg, 84%).^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.94 (t, ^3J = 7.4 Hz, 3H, CH_3), 1.33-1.50 (m, 2H, CH_2), 1.61-1.74 (m, 2H, CH_2), 3.73 (s, 3H, OCH_3), 4.34 (t, ^3J = 6.7 Hz, 2H, CH_2), 6.82-6.87 (m, 1H, Ar), 7.02-7.05 (dd, ^3J = 8.4 Hz, ^4J = 0.8 Hz, 1H, Ar), 7.11 (d, ^3J = 8.7 Hz, 1H, Ar), 7.37-7.40 (dd, ^3J = 8.1 Hz, ^4J = 1.6 Hz, 1H, Ar), 7.46-7.52 (m, 1H, Ar), 7.54 (d, ^3J = 8.6 Hz, 1H, Ar), 11.27 (s, 1H, OH), 11.64 (s, 1H, OH). ^{13}C NMR (75 MHz, $CDCl_3$): δ = 13.5 (CH_3), 18.8, 30.1 (CH_2), 52.6 (OCH_3), 66.7 (CH_2), 111.0 (C), 118.3, 118.4, 118.8 (Ar), 119.0, 128.2 (C), 133.1, 135.1 (Ar), 136.3 (C), 136.7 (Ar), 163.0, 163.1, 167.4, 168.7, 199.7 (C). IR (ATR, cm^{-1}): $\tilde{\nu}$ = 2958 (w), 2874 (w), 1736 (s), 1673 (s), 1625 (s), 1608 (m), 1581 (s), 1442 (s), 1325 (s), 1211(s), 1146 (s), 1018 (s), 756 (s) , 641 (s). GC-MS (EI, 70 eV): *m/z* (%) = 372 (M^+, 7), 340 (47), 284 (21), 283 (34), 239 (100), 212 (22). HRMS (EI, 70 eV): calcd. for $C_{20}H_{20}O_7$ (M^+) 372.12035, found 372.12116. Anal. calcd. for $C_{20}H_{20}O_7$ (372.37): C, 64.51; H, 4.41. Found: C, 64.28; H, 5.54.

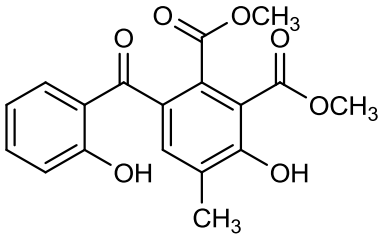
1-Methyl 2-octyl 3-hydroxy-6-(2-hydroxybenzoyl)phthalate (8c).



Starting with 3-methoxalylchromone (**3a**) (0.232 g, 1.0 mmol) and 1-octyloxy-1,3-bis(trimethylsilyloxy)-1,3-butadiene (**5h**) (0.717 g, 2.0 mmol), Me_3SiOTf (0.36 mL, 2.0 mmol) in CH_2Cl_2 (4 mL) and then 10 h heating in EtOH (4 mL) with 3 mol% of p-TsOH, the product **8c** was isolated as a yellow oil (0.342 mg, 80%).^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.86 (t, ^3J = 6.7 Hz, 3H, CH_3), 1.26-1.42 (m, 10H, $(CH_2)_5$), 1.66-1.76 (m, 2H, CH_2), 3.74 (s, 3H, OCH_3), 4.34 (t, ^3J = 6.8 Hz, 2H, CH_2), 6.83-6.88 (m, 1H, Ar), 7.03-7.06 (dd, ^3J = 8.4 Hz, ^4J = 0.8 Hz, 1H, Ar), 7.11 (d, ^3J = 8.7 Hz, 1H, Ar), 7.37-7.41 (dd, ^3J = 8.0 Hz, ^4J = 1.5 Hz, 1H, Ar), 7.47-7.53 (m, 1H, Ar), 7.55 (d, ^3J = 8.6 Hz, 1H, Ar), 11.28 (s, 1H, OH), 11.65 (s, 1H,

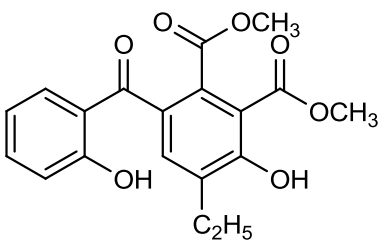
OH). ^{13}C NMR (75 MHz, $CDCl_3$): δ = 14.0 (CH_3), 22.5, 25.7, 28.2, 29.1, 29.2, 31.7 (CH_2), 52.6 (OCH_3), 67.1 (CH_2), 111.1 (C), 118.4, 118.8 (Ar), 119.1, 128.3 (C), 133.1, 135.2 (Ar), 136.4 (C), 136.8 (Ar), 163.0, 163.3, 167.4, 168.7, 199.7 (C). IR (ATR, cm^{-1}): $\tilde{\nu}$ = 2925 (m), 2855 (m), 1738 (s), 1674 (s), 1627 (s), 1609 (m), 1583 (s), 1443 (s), 1329 (s), 1214(s), 1147 (s), 1018 (s), 757 (s) , 642 (s). GC-MS (EI, 70 eV): *m/z* (%) = 428 (M^+, 4), 396 (47), 284 (44), 283 (42), 239 (100), 212 (22). HRMS (ESI): calcd. for $C_{24}H_{29}O_7$ ($[M+H]^+$) 429.1902, found 429.1902; calcd. for $C_{24}H_{28}NaO_7$ ($[M+Na]^+$) 451.1727, found 451.1730. Anal. calcd. for $C_{24}H_{28}O_7$ (428.47): C, 67.28; H, 6.59. Found: C, 67.23; H, 7.42.

Dimethyl 3-hydroxy-6-(2-hydroxybenzoyl)-4-methylphthalate (8d).



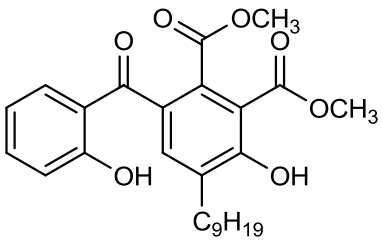
Starting with 3-methoxalylchromone (**3a**) (0.232 g, 1.0 mmol) and 1-methoxy-1,3-bis(trimethylsilyloxy)-1,3-pentadiene (**5i**) (0.549 g, 2.0 mmol), Me_3SiOTf (0.36 mL, 2.0 mmol) in CH_2Cl_2 (4 mL) and then 10 h heating in EtOH (4 mL) with 3 mol% of p-TsOH, the product **8d** was isolated as a slight yellow solid (0.230 mg, 67%); mp = 98-100 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 2.31 (s, 3H, CH_3), 3.73 (s, 3H, OCH_3), 3.93 (s, 3H, OCH_3), 6.82-6.88 (m, 1H, Ar), 7.02-7.05 (dd, ^3J = 8.4 Hz, ^4J = 0.8 Hz, 1H, Ar), 7.38-7.42 (m, 2H, Ar), 7.47-7.53 (m, 1H, Ar), 11.30 (s, 1H, OH), 11.66 (s, 1H, OH). ^{13}C NMR (75 MHz, $CDCl_3$): δ = 16.0 (CH_3), 52.6, 53.2 (OCH_3), 111.0 (C), 118.3, 118.8 (Ar), 119.2, 127.8, 128.23 (C), 133.1 (Ar), 133.8 (C), 135.4, 136.6 (Ar), 161.5, 162.9, 167.7, 169.4, 200.0 (C). IR (ATR, cm^{-1}): $\tilde{\nu}$ = 3004 (w), 2951 (w), 2850 (w), 1728 (s), 1676 (s), 1622 (s), 1601 (s), 1577 (s), 1438 (s), 1349 (s), 1253 (s), 1151 (s), 1048 (s), 980 (s), 762 (s), 662 (s). GC-MS (EI, 70 eV): *m/z* (%) = 344 (M^+, 5), 312 (35), 280 (32), 253 (100). HRMS (EI, 70 eV): calcd. for $C_{18}H_{16}O_7$ (M^+) 344.08905, found 344.09011. Anal. calcd. for $C_{18}H_{16}O_7$ (344.32): C, 62.79; H, 4.64. Found: C, 62.70; H, 4.69.

Dimethyl 5-ethyl-3-(2-hydroxybenzoyl)phthalate (8e).



Starting with 3-methoxalylchromone (**3a**) (0.232 g, 1.0 mmol) and 1-methoxy-1,3-bis(trimethylsilyloxy)-1,3-hexadiene (**5j**) (0.577 g, 2.0 mmol), Me_3SiOTf (0.36 mL, 2.0 mmol) in CH_2Cl_2 (4 mL) and then 10 h heating in EtOH (4 mL) with 3 mol% of p-TsOH, the product **8e** was isolated as a slight yellow solid (0.237 mg, 66%); mp = 126-128 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.21 (t, ^3J = 7.4 Hz , 3H, CH_3), 2.73 (q, ^3J = 7.4 Hz, 2H, CH_2), 3.74 (s, 3H, OCH_3), 3.93 (s, 3H, OCH_3), 6.83-6.88 (m, 1H, Ar), 7.03-7.06 (dd, ^3J = 8.4 Hz, ^4J = 0.8 Hz, 1H, Ar), 7.37-7.42 (m, 2H, Ar), 7.47-7.53 (m, 1H, Ar), 11.31 (s, 1H, OH), 11.68 (s, 1H, OH). ^{13}C NMR (75 MHz, $CDCl_3$): δ = 13.2 (CH_3), 22.9 (CH_2), 52.6, 53.2 (OCH_3), 111.3 (C), 118.3, 118.7 (Ar), 119.2, 127.9 (C), 133.1 (Ar), 133.7 (C), 133.9 (Ar), 134.0 (C), 136.6 (Ar), 161.2, 163.0, 167.8, 169.5, 200.1 (C). IR (ATR, cm^{-1}): $\tilde{\nu}$ = 2973 (w), 2954 (w), 1725 (s), 1675 (s), 1624 (s), 1599 (s), 1575 (m), 1446 (s), 1359 (s), 1248 (s), 1154 (s), 755 (s), 716 (s), 657 (s), 384 (s). GC-MS (EI, 70 eV): *m/z* (%) = 358 (M^+, 5), 326 (37), 294 (24), 267 (100). HRMS (ESI): calcd. for $C_{19}H_{19}O_7$ ($[M+H]^+$) 359.1125, found 359.1122; calcd. for $C_{19}H_{18}NaO_7$ ($[M+Na]^+$) 381.0945, found 381.0943. Anal. calcd. for $C_{19}H_{18}O_7$ (358.34): C, 63.68; H, 5.06. Found: C, 63.71; H, 5.22.

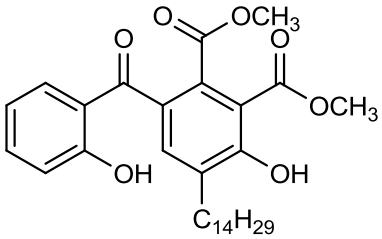
Dimethyl 3-(2-hydroxybenzoyl)-5-nonylphthalate (8f).



Starting with 3-methoxalylchromone (**3a**) (0.232 g, 1.0 mmol) and 1-methoxy-1,3-bis(trimethylsilyloxy)-1,3-tridecadiene (**5k**) (0.773 g, 2.0 mmol), Me_3SiOTf (0.36 mL, 2.0 mmol) in CH_2Cl_2 (4 mL) and then 10 h heating in EtOH (4 mL) with 3 mol% of p-TsOH, the product **8f** was isolated as a slight yellow oil (0.318 mg, 70%). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.86 (t, ^3J = 6.8 Hz , 3H, CH_3), 1.21-1.29 (m, 12H, $(CH_2)_6$), 1.54-1.61 (m, 2H, CH_2), 2.68 (t, ^3J = 7.5 Hz, 2H, CH_2), 3.75 (s, 3H, OCH_3), 3.93 (s, 3H, OCH_3), 6.82-6.88 (m, 1H, Ar), 7.03-7.06 (dd, ^3J = 8.4 Hz, ^4J = 1.0 Hz, 1H, Ar), 7.38-7.41 (m, 2H, Ar), 7.48-7.53 (m, 1H, Ar), 11.31 (s, 1H, OH), 11.68 (s, 1H, OH). ^{13}C NMR (75 MHz, $CDCl_3$): δ = 14.0 (CH_3), 22.6, 28.9, 29.2, 29.3, 29.4, 29.5, 29.7, 31.8 (CH_2), 52.7, 53.2 (OCH_3), 110.4 (C), 118.3, 118.7 (Ar), 119.2, 127.7, 132.7 (C), 133.1 (Ar), 133.8 (C), 134.8, 136.6 (Ar), 161.3, 163.0, 167.8, 169.5, 200.1 (C). IR (ATR, cm^{-1}): $\tilde{\nu}$ = 3074 (w), 2948 (m), 2925 (s), 2853 (m), 1739 (s), 1685 (s), 1625 (s), 1610 (m), 1577 (m), 1439 (s), 1359 (s), 1241 (s), 1152 (s), 1057 (s), 770 (s). MS (EI, 70 eV): *m/z* (%) = 456

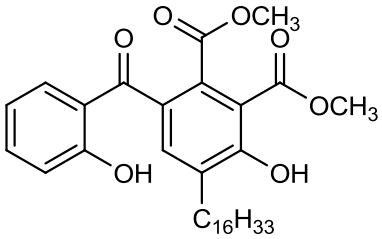
(M^+, 10), 425 (41), 424 (90), 397 (46), 393 (39), 392 (98), 366 (34), 365 (100), 281 (32), 280 (89), 252 (25), 121 (32), 84 (28), 83 (23), 71 (27), 69 (30), 57 (48), 55 (39), 44 (38), 43 (65), 41 (43). HRMS (EI, 70 eV): calcd. for $C_{26}H_{32}O_7$ (M^+) 456.21425, found 456.21447. Anal. calcd. for $C_{29}H_{32}O_7$ (456.53): C, 68.40; H, 7.07. Found: C, 68.88; H, 7.08.

Dimethyl 3-(2-hydroxybenzoyl)-5-tetradecylphthalate (8g).



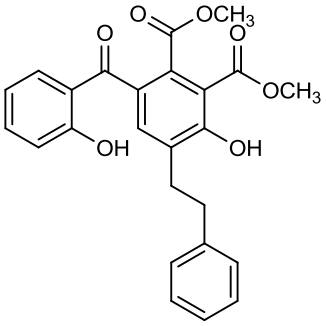
Starting with 3-methoxalylchromone (**3a**) (0.232 g, 1.0 mmol) and 1-methoxy-1,3-bis(trimethylsilyloxy)-1,3-octadecadiene (**5l**) (0.913 g, 2.0 mmol), Me_3SiOTf (0.36 mL, 2.0 mmol) in CH_2Cl_2 (4 mL) and then10 h heating in EtOH (4 mL) with 3 mol% of p-TsOH, the product **8g** was isolated as a yellow solid (0.390 mg, 74%); mp = 82-84 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.87 (t, ^3J = 6.7 Hz , 3H, CH_3), 1.24-1.29 (m, 22H, $(CH_2)_{11}$), 1.54-1.64 (m, 2H, CH_2), 2.68 (t, ^3J = 7.5 Hz, 2H, CH_2), 3.75 (s, 3H, OCH_3), 3.93 (s, 3H, OCH_3), 6.82-6.88 (m, 1H, Ar), 7.03-7.06 (dd, ^3J = 8.3 Hz, ^4J = 0.8 Hz, 1H, Ar), 7.38-7.41 (m, 2H, Ar), 7.47-7.53 (m, 1H, Ar), 11.31 (s, 1H, OH), 11.68 (s, 1H, OH). ^{13}C NMR (75 MHz, $CDCl_3$): δ = 14.1 (CH_3), 22.6, 28.9, 29.2, 29.3, 29.4, 29.5, 29.6, 29.7, 31.9 (CH_2), 52.6, 53.2 (OCH_3), 110.4 (C), 118.3, 118.7 (Ar), 119.2, 127.7, 132.7 (C), 133.1 (Ar), 133.8 (C), 134.8, 136.6 (Ar), 161.3, 163.0, 167.8, 169.5, 200.1 (C). IR (ATR, cm^{-1}): $\tilde{\nu}$ = 2953 (w), 2916 (s), 2848 (m), 1729 (s), 1674 (s), 1627 (s), 1602 (m), 1576 (m), 1446 (s), 1359 (s), 1269 (s), 1211 (s), 1155 (s), 766 (s), 723 (s), 664 (s). MS (EI, 70 eV): *m/z* (%) = 526 (M^+, 3), 495 (34), 494 (100), 467 (30), 463 (24), 462 (76), 453 (55), 282 (22), 280 (62), 121 (21), 44 (40), 43 (26), 41 (20). HRMS (EI, 70 eV): calcd. for $C_{31}H_{42}O_7$ (M^+) 526.29251, found 526.29356. Anal. calcd. for $C_{31}H_{42}O_7$ (526.66): C, 70.70; H, 8.04. Found: C, 70.86; H, 8.06.

Dimethyl 5-hexadecyl-3-(2-hydroxybenzoyl)phthalate (8h).



Starting with 3-methoxalylchromone (**3a**) (0.232 g, 1.0 mmol) and 1-methoxy-1,3-bis(trimethylsilyloxy)-1,3-icosadiene (**5m**) (0.969 g, 2.0 mmol), Me_3SiOTf (0.36 mL, 2.0 mmol) in CH_2Cl_2 (4 mL) and then 10 h heating in EtOH (3 mL) with 3 mol% of p-TsOH, the product **8h** was isolated as a slight yellow solid (0.399 mg, 72%); mp = 78-80 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.87 (t, ^3J = 6.6 Hz , 3H, CH_3), 1.24-1.29 (m, 26H, $(CH_2)_{13}$), 1.54-1.61 (m, 2H, CH_2), 2.68 (t, ^3J = 7.5 Hz, 2H, CH_2), 3.75 (s, 3H, OCH_3), 3.93 (s, 3H, OCH_3), 6.82-6.88 (m, 1H, Ar), 7.03-7.06 (dd, ^3J = 8.4 Hz, ^4J = 0.8 Hz, 1H, Ar), 7.38-7.41 (m, 2H, Ar), 7.47-7.53 (m, 1H, Ar), 11.31 (s, 1H, OH), 11.68 (s, 1H, OH). ^{13}C NMR (75 MHz, $CDCl_3$): δ = 14.1 (CH_3), 22.6, 28.9, 29.3, 29.4, 29.5, 29.6, 29.7, 31.9 (CH_2), 52.7, 53.2 (OCH_3), 110.4 (C), 118.3, 118.7 (Ar), 119.2, 127.7, 132.7 (C), 133.1 (Ar), 133.8 (C), 134.8, 136.6 (Ar), 161.3, 163.0, 167.8, 169.5, 200.1 (C). IR (ATR, cm^{-1}): $\tilde{\nu}$ = 2916 (s), 2848 (s), 1741 (s), 1686 (s), 1624 (s), 1608 (m), 1577 (m), 1439 (s), 1359 (s), 1254 (s), 1221 (s), 1198 (s), 1154 (s), 1057 (s), 759 (s), 600 (s). MS (EI, 70 eV): *m/z* (%) = 554 (M^+, 4), 523 (41), 522 (100), 495 (33), 490 (62), 463 (36), 280 (34), 69 (27), 55 (28), 44 (92), 43 (51), 41 (27). HRMS (EI, 70 eV): calcd. for $C_{33}H_{46}O_7$ (M^+) 554.32381, found 554.32305. Anal. calcd. for $C_{33}H_{46}O_7$ (554.71): C, 71.45; H, 8.36. Found: C, 71.58; H, 8.65.

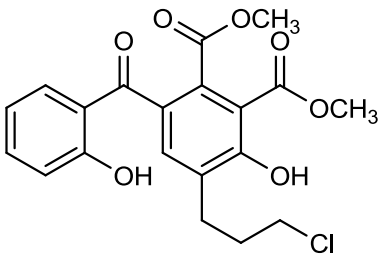
Dimethyl 3-(2-hydroxybenzoyl)-5-phenethylphthalate (8i).



Starting with 3-methoxalylchromone (**3a**) (0.232 g, 1.0 mmol) and 1-methoxy-6-phenyl-1,3-bis(trimethylsilyloxy)-1,3-hexadiene (**5n**) (0.729 g, 2.0 mmol), Me_3SiOTf (0.36 mL, 2.0 mmol) in CH_2Cl_2 (4 mL) and then 10 h heating in EtOH (4 mL) with 3 mol% of p-TsOH, the product **8i** was isolated as a yellow solid (0.161 mg, 37%); mp = 129-131 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 2.98-3.10 (m, 24H, $(CH_2)_2$), 3.82 (s, 3H, OCH_3), 4.01 (s, 3H, OCH_3), 6.79-6.84 (m, 1H, Ar), 7.06-7.36 (m, 8H, Ar), 7.50-7.56 (m, 1H, Ar), 11.48 (s, 1H, OH), 11.73 (s, 1H, OH). ^{13}C NMR (75 MHz, $CDCl_3$): δ = 32.2, 34.6 (CH_2), 52.7, 53.3 (OCH_3), 110.5 (C), 118.2, 118.9 (Ar), 119.0 (C), 126.0 (Ar), 127.6 (C), 128.4, 128.5 (Ar), 131.0 (C), 133.1 (Ar), 134.3 (C), 135.4, 136.6 (Ar), 161.4, 163.0, 167.8, 169.5, 199.8 (C). IR (ATR, cm^{-1}): $\tilde{\nu}$ = 3032 (w), 2952 (w), 1723 (s), 1669 (s), 1622 (s), 1594 (s), 1574 (s), 1434 (s), 1344 (s), 1267 (s), 1220 (s), 1148 (s),

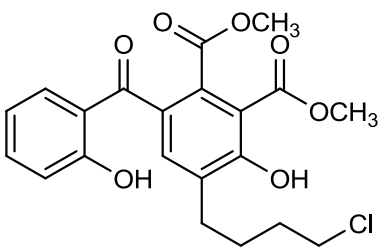
1052 (s), 975 (s), 815 (s), 763 (s), 698 (s). MS (EI, 70 eV): *m/z* (%) = 434 (M^+, 13), 403 (24), 402 (66), 375 (28), 371 (25), 370 (100), 343 (78), 279 (38), 251 (48), 91 (55). HRMS (EI, 70 eV): calcd. for $C_{25}H_{22}O_7$ (M^+) 434.13600, found 434.13619. Anal. calcd. for $C_{25}H_{22}O_7$ (434.44): C, 69.12; H, 5.10. Found: C, 69.15; H, 4.99.

Dimethyl 4-(3-chloropropyl)-3-hydroxy-6-(2-hydroxybenzoyl)phthalate (8j).



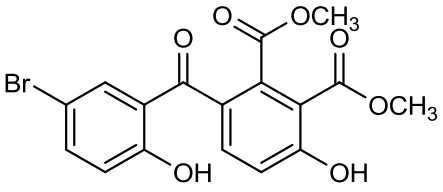
Starting with 3-methoxalylchromone (**3a**) (0.232 g, 1.0 mmol) and 1-methoxy-7-chlor-1,3-bis(trimethylsilyloxy)-1,3-pentadiene (**5o**) (0.674 g, 2.0 mmol), Me_3SiOTf (0.36 mL, 2.0 mmol) in CH_2Cl_2 (4 mL), the precipitated product **8j** was isolated as a slight yellow solid (0.216 mg, 53%); mp = 160-161 °C. ^1H NMR (300 MHz, $CDCl_3$): δ = 2.06-2.15 (m, 2H, CH_2), 2.88 (t, ^3J = 7.4 Hz , 2H, CH_2), 3.54 (t, ^3J = 6.3 Hz , 2H, CH_2), 3.75 (s, 3H, OCH_3), 3.94 (s, 3H, OCH_3), 6.83-6.89 (m, 1H, Ar), 7.03-7.06 (dd, ^3J = 8.4 Hz, ^4J = 0.9 Hz, 1H, Ar), 7.37-7.40 (dd, ^3J = 8.0 Hz, ^4J = 1.5 Hz, 1H, Ar), 7.46 (s, 1H, Ar), 7.48-7.54 (m, 1H, Ar), 11.38 (s, 1H, OH), 11.65 (s, 1H, OH). ^{13}C NMR (75 MHz, $CDCl_3$): δ = 27.4, 31.2, 44.2 (CH_2), 52.7, 53.3 (OCH_3), 110.6 (C), 118.4, 118.8 (Ar), 119.1 (C), 127.9, 130.5 (C), 133.1 (Ar), 134.6 (C), 135.3, 136.8 (Ar), 161.3, 163.0, 167.6, 169.4, 199.8 (C). IR (ATR, cm^{-1}): $\tilde{\nu}$ = 3168 (w), 3002 (w), 2954 (w), 2849 (w), 1723 (s), 1677 (s), 1622 (s), 1595 (s), 1576 (s), 1436 (s), 1255 (s), 1154 (s), 1053 (s), 979 (s), 816 (s), 770 (s), 633 (s). MS (EI, 70 eV): *m/z* (%) = 406 (M^+, 7), 374 (53), 317 (34), 315 (100), 307 (56), 280 (49), 279 (90), 69 (31), 57 (33), 43 (29). HRMS (ESI): calcd. for $C_{20}H_{20}Cl^{35}O_7$ ($[M+H]^+$) 407.0892, found 407.0888; calcd. for $C_{20}H_{19}Cl^{35}NaO_7$ ($[M+Na]^+$) 429.0712, found 429.0712; calcd. for $C_{20}H_{19}Cl^{37}NaO_7$ ($[M+Na]^+$) 431.0691, found 431.0690. Anal. calcd. for $C_{20}H_{19}ClO_7$ (406.08): C, 59.05; H, 4.71. Found: C, 59.18; H, 4.65.

Dimethyl 4-(3-chloropropyl)-3-hydroxy-6-(2-hydroxybenzoyl)phthalate (8k).



Starting with 3-methoxalylchromone (**3a**) (0.232 g, 1.0 mmol) and 1-methoxy-8-chlor-1,3-bis(trimethylsilyloxy)-1,3-hexadiene (**5p**) (0.702 g, 2.0 mmol), Me_3SiOTf (0.36 mL, 2.0 mmol) in CH_2Cl_2 (4 mL), and then 10 h heating in EtOH (4 mL) with 3 mol% of p-TsOH, the product **8k** was isolated as a yellow solid (0.237 mg, 56%); mp = 87-88 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.71-1.87 (m, 4H, $(CH_2)_2$), 2.73 (t, ^3J = 7.0 Hz , 2H, CH_2), 3.55 (t, ^3J = 6.3 Hz , 2H, CH_2), 3.74 (s, 3H, OCH_3), 3.94 (s, 3H, OCH_3), 6.83-6.89 (m, 1H, Ar), 7.03-7.06 (dd, ^3J = 8.4 Hz, ^4J = 0.9 Hz, 1H, Ar), 7.36-7.41 (dd, ^3J = 8.0 Hz, ^4J = 1.6 Hz, 1H, Ar), 7.41 (s, 1H, Ar), 7.48-7.54 (m, 1H, Ar), 11.34 (s, 1H, OH), 11.65 (s, 1H, OH). ^{13}C NMR (75 MHz, $CDCl_3$): δ = 26.2, 28.9, 32.1, 44.6 (CH_2), 52.7, 53.3 (OCH_3), 110.5 (C), 118.4, 118.8 (Ar), 119.2 (C), 127.9, 131.8 (C), 133.1 (Ar), 134.1 (C), 134.8, 136.7 (Ar), 161.2, 163.0, 167.7, 169.4, 199.9 (C). IR (ATR, cm^{-1}): $\tilde{\nu}$ = 2994 (w), 2949 (m), 2927 (m), 2898 (m), 1723 (s), 1675 (s), 1623 (s), 1597 (s), 1547 (s), 1437 (s), 1348 (s), 1223 (s), 1151 (s), 1054 (s), 762 (s), 742 (s), 648 (s), 634 (s). MS (EI, 70 eV): *m/z* (%) = 420 (M^+, 10), 390 (38), 389 (33), 388 (85), 361 (26), 331 (66), 330 (36), 329 (100), 321 (34), 293 (73), 121 (26). HRMS (EI, 70 eV): calcd. for $C_{21}H_{21}O_7Cl$ (M^+) 420.09703, found 420.09622. Anal. calcd. for $C_{21}H_{21}ClO_7$ (420.84): C, 59.93; H, 5.03. Found: C, 60.87; H, 5.10.

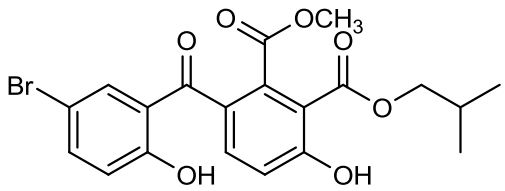
Dimethyl 3-(5-bromo-2-hydroxybenzoyl)-6-hydroxyphthalate (8l).



Starting with 6-brom-3-methoxalylchromone (**3b**) (0.309 g, 1.0 mmol) and 1-methoxy-1,3-bis(trimethylsilyloxy)-1,3-butadiene (**5a**) (0.520 g, 2.0 mmol), Me_3SiOTf (0.36 mL, 2.0 mmol) in CH_2Cl_2 (8 mL), and then 5 h heating in EtOH (4 mL) with 3 mol% of p-TsOH, the product **8l** was isolated as a yellow solid (0.200 mg, 49%); mp = 112-118 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 3.83 (s, 3H, OCH_3), 4.00 (s, 3H, OCH_3), 7.01 (d, ^3J = 8.8 Hz, 1H, Ar), 7.21 (d, ^3J = 8.7 Hz, 1H, Ar), 7.53 (d, ^4J = 2.4 Hz, 1H, Ar), 7.59-7.64 (m, 2H, Ar), 11.18 (s, 1H, OH), 11.58 (s, 1H, OH). ^{13}C NMR (75 MHz, $CDCl_3$): δ = 52.8, 53.4 (OCH_3), 110.4, 111.1 (C), 118.7 (Ar), 120.3 (C), 120.4 (Ar), 127.6 (C), 134.9, 135.1 (Ar), 136.6 (C), 139.4 (Ar), 161.9, 163.4, 167.4, 168.8, 198.6 (C). IR (ATR, cm^{-1}): $\tilde{\nu}$ = 3305 (w), 2950 (w), 2927 (m), 1738 (m), 1686 (m), 1628 (m), 1590 (m), 1464 (m), 1434 (m), 1184 (s), 1144 (s), 1120 (s), 1019 (s), 942 (s), 648 (s), 525 (s). MS (EI, 70 eV): *m/z*

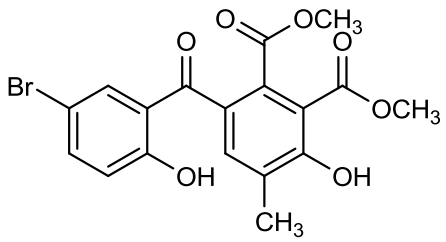
(%) = 409 (M^+, 2), 378 (46), 376 (46), 319 (99), 318 (33), 317 (100). HRMS (ESI): calcd. for $C_{17}H_{13}Br^{79}NaO_7$ ([M+Na]$^+$) 430.9736, found 430.9728; calcd. for $C_{17}H_{13}Br^{81}NaO_7$ ([M+Na]$^+$) 432.9718, found 432.97135. Anal. calcd. for $C_{17}H_{13}BrO_7$ (409.18): C, 49.90; H, 3.20. Found: C, 49.66; H, 3.44.

1-Isobutyl 2-methyl 3-(5-bromo-2-hydroxybenzoyl)-6-hydroxyphthalate (8m).



Starting with 6-brom-3-methoxalylchromone (**3b**) (0.309 g, 1.0 mmol) and 1-isobutoxy-1,3-bis(trimethylsilyloxy)-1,3-butadiene (**5f**) (0.605 g, 2.0 mmol), Me_3SiOTf (0.36 mL, 2.0 mmol) in CH_2Cl_2 (4 mL), and then 5 h heating in EtOH (8 mL) with 3 mol% of p-TsOH, the product **8m** was isolated as a yellow solid (0.325 mg, 72%); mp = 106-108 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.96 (s, 3H, CH_3), 0.98 (s, 3H, CH_3), 1.95-2.08 (m, 1H, CH), 3.74 (s, 3H, OCH_3), 4.14 (d, ^3J = 6.9 Hz, 2H, CH_2), 6.96 (d, ^3J = 8.8 Hz, 1H, Ar), 7.15 (d, ^3J = 8.7 Hz, 1H, Ar), 7.49-7.59 (m, 3H, Ar), 11.32 (s, 1H, OH), 11.56 (s, 1H, OH). ^{13}C NMR (75 MHz, $CDCl_3$): δ = 19.0 ($(CH_3)_2$), 27.6 (CH), 52.7 (OCH_3), 73.0 (CH_2), 110.4, 111.2 (C), 118.7 (Ar), 120.3 (C), 120.4 (Ar), 127.6 (C), 134.9 (Ar), 136.4 (C), 139.4 (Ar), 161.9, 163.6, 167.2, 168.6, 198.8 (C). IR (ATR, cm^{-1}): $\tilde{\nu}$ = 2959 (w), 1724 (s), 1669 (m), 1625 (m), 1602 (m), 1581 (m), 1464 (s), 1321 (s), 1221 (s), 1147 (s), 1018 (s), 624 (s). MS (EI, 70 eV): *m/z* (%) = 451 (M^+, 2), 420 (46), 418 (45), 364 (100), 363 (29), 362 (96), 319 (70), 318 (21), 317 (69) 57 (29). Anal. calcd. for $C_{20}H_{19}BrO_7$ (451.26): C, 53.23; H, 4.24. Found: C, 53.37; H, 4.42.

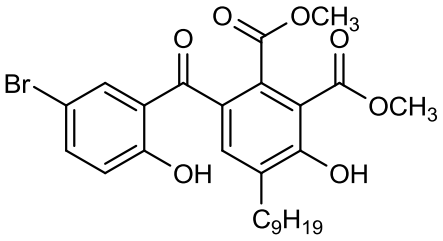
Dimethyl 6-(5-bromo-2-hydroxybenzoyl)-3-hydroxy-4-methylphthalate (8n).



Starting with 6-brom-3-methoxalylchromone (**3b**) (0.309 g, 1.0 mmol) and 1-methoxy-1,3-bis(trimethylsilyloxy)-1,3-pentadiene (**5i**) (0.549 g, 2.0 mmol), Me_3SiOTf (0.36 mL, 2.0 mmol) in CH_2Cl_2 (4 mL), and then 5 h heating in EtOH (8 mL) with 3 mol% of p-TsOH, the product **8n** was isolated as a yellow solid (0.318 mg, 75%); mp = 150-153 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 2.33 (s, 3H, CH_3), 3.73

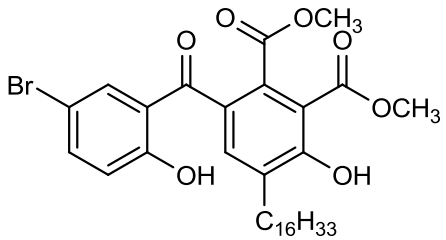
(s, 3H, OCH_3), 3.94 (s, 3H, OCH_3), 6.95 (d, ^3J = 8.8 Hz, 1H, Ar), 7.39 (bs, 1H, Ar), 7.48 (d, ^4J = 2.4 Hz, 1H, Ar), 7.57 (dd, ^3J = 8.8 Hz, ^4J = 2.4 Hz, 1H, Ar), 11.33 (s, 1H, OH), 11.56 (s, 1H, OH). ^{13}C NMR (75 MHz, $CDCl_3$): δ = 16.0 (CH_3), 52.7, 53.3 (OCH_3), 110.3, 110.4 (C), 120.4 (Ar), 120.5, 127.2, 128.5 (C), 134.9, 135.0, 139.3 (Ar), 161.8, 161.9, 167.7, 169.4, 199.2 (C). IR (ATR, cm^{-1}): $\tilde{\nu}$ = 2954 (w), 1734 (s), 1673 (m), 1630 (m), 1595 (m), 1464 (m), 1357 (s), 1255 (s), 1158 (s), 1053 (s), 990 (s), 803 (s), 683 (s), 625 (s), 416 (s). MS (EI, 70 eV): *m/z* (%) = 423 (M^+, 2), 392 (52), 390 (50), 360 (36), 358 (34), 333 (96), 332 (33), 331 (100). HRMS (EI, 70 eV): calcd. for $C_{18}H_{15}O_7Br^{79}$ (M^+) 421.99957, found 421.99850; calcd. for $C_{18}H_{15}O_7Br^{81}$ (M^+) 423.9975, found 423.99705. Anal. calcd. for $C_{18}H_{15}BrO_7$ (423.21): C, 51.08; H, 3.57. Found: C, 51.17; H, 3.66.

Dimethyl 6-(5-bromo-2-hydroxybenzoyl)-3-hydroxy-4-nonylphthalate (8o).



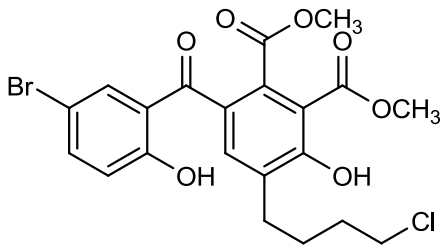
Starting with 6-brom-3-methoxalylchromone **(3b)** (0.309 g, 1.0 mmol) and 1-methoxy-1,3-bis(trimethylsilyloxy)-1,3-tridecadiene (**5k**) (0.773 g, 2.0 mmol), Me_3SiOTf (0.36 mL, 2.0 mmol) in CH_2Cl_2 (4 mL), and then 5 h heating in EtOH (8 mL) with 3 mol% of p-TsOH, the product **8o** was isolated as a yellow oil (0.414 mg, 77%).^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.86 (t, ^3J = 6.6 Hz, 3H, CH_3), 1.25-1.36 (m, 12H, $(CH_2)_6$), 1.56-1.66 (m, 2H, CH_2), 2.70 (t, ^3J = 7.5 Hz, 2H, CH_2), 3.76 (s, 3H, OCH_3), 3.94 (s, 3H, OCH_3), 6.96 (d, ^3J = 8.8 Hz, 1H, Ar), 7.38 (bs, 1H, Ar), 7.50 (d, ^4J = 2.4 Hz, 1H, Ar), 7.57 (dd, ^3J = 8.8 Hz, ^4J = 2.4 Hz, 1H, Ar), 11.36 (s, 1H, OH), 11.59 (s, 1H, OH). ^{13}C NMR (75 MHz, $CDCl_3$): δ = 14.1 (CH_3), 22.6, 28.9, 29.3, 29.4, 29.7, 31.8 (CH_2), 52.7, 53.3 (OCH_3), 110.3, 110.5 (C), 120.4 (Ar), 120.5, 127.0, 133.1, 133.9 (C), 134.6, 135.1, 139.2 (Ar), 161.6, 161.9, 167.7, 169.4, 199.1 (C). IR (ATR, cm^{-1}): $\tilde{\nu}$ = 3068 (w), 2953 (w), 2920 (s), 2853 (m), 1729 (s), 1688 (m), 1634 (s), 1610 (m), 1438 (s), 1264 (s), 1227 (s), 1192 (s), 1167 (s), 975 (s), 811 (s), 713 (s), 695 (s). MS (EI, 70 eV): *m/z* (%) = 535 (M^+, 2), 505 (28), 504 (100), 503 (29), 502 (94), 445 (78), 444 (17), 443 (76), 360 (46), 359 (13), 358 (45). HRMS (ESI): calcd. for $C_{26}H_{31}Br^{79}NaO_7$ ($[M+Na]^+$) 557.1145, found 557.1140; calcd. for $C_{26}H_{31}Br^{81}NaO_7$ ($[M+Na]^+$) 559.1129, found 559.1144. Anal. calcd. for $C_{26}H_{31}BrO_7$ (535.42): C, 58.32; H, 5.84. Found: C, 57.06; H, 6.33.

Dimethyl 6-(5-bromo-2-hydroxybenzoyl)-4-hexadecyl-3-hydroxyphthalate (8p).



Starting with 6-brom-3-methoxalylchromone **(3b)** (0.309 g, 1.0 mmol) and 1-methoxy-1,3-bis(trimethylsilyloxy)-1,3-icosadiene (**5m**) (0.969 g, 2.0 mmol), Me_3SiOTf (0.36 mL, 2.0 mmol) in CH_2Cl_2 (4 mL), and then 5 h heating in EtOH (8 mL) with 3 mol% of p-TsOH, the product **8p** was isolated as a yellow solid (0.340 mg, 54%); mp = 82-83 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.80 (t, ^3J = 6.7 Hz, 3H, CH_3), 1.17-1.24 (m, 26H, $(CH_2)_{13}$), 1.49-1.59 (m, 2H, CH_2), 2.63 (t, ^3J = 7.5 Hz, 2H, CH_2), 3.69 (s, 3H, OCH_3), 3.87 (s, 3H, OCH_3), 6.89 (d, ^3J = 8.8 Hz, 1H, Ar), 7.31 (bs, 1H, Ar), 7.43 (d, ^4J = 2.4 Hz, 1H, Ar), 7.50 (dd, ^3J = 8.8 Hz, ^4J = 2.4 Hz, 1H, Ar), 11.29 (s, 1H, OH), 11.52 (s, 1H, OH). ^{13}C NMR (75 MHz, $CDCl_3$): δ = 14.1 (CH_3), 22.6, 28.9, 29.3, 29.4, 29.5, 29.6, 29.7, 29.8, 31.9 (CH_2), 52.7, 53.3 (OCH_3), 110.3, 110.5 (C), 120.4 (Ar), 120.5, 127.0, 133.1, 133.9 (C), 134.6, 135.0, 139.2 (Ar), 161.6, 161.9, 167.7, 169.4, 199.1 (C). IR (ATR, cm^{-1}): $\tilde{\nu}$ = 2955 (w), 2919 (s), 2852 (s), 1731 (s), 1673 (s), 1634 (s), 1608 (w), 1583 (w), 1435 (s), 1264 (s), 1226 (s), 1208 (s), 1191 (s), 1170 (s), 979 (s), 701 (s), 692 (s). MS (EI, 70 eV): *m/z* (%) = 633 (M^+, 1), 603 (32), 602 (100), 601 (32), 600 (93), 570 (11), 568 (10), 543 (19), 541 (18), 360 (17), 358 (16). HRMS (ESI): calcd. for $C_{33}H_{45}Br^{79}NaO_7$ ($[M+Na]^+$) 655.2240, found 655.2238; calcd. for $C_{33}H_{45}Br^{81}NaO_7$ ($[M+Na]^+$) 657.2226, found 657.2229. Anal. calcd. for $C_{33}H_{43}BrO_7$ (633.61): C, 62.55; H, 7.16. Found: C, 62.56; H, 7.47.

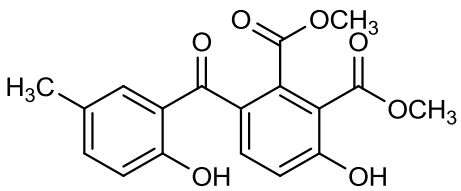
Dimethyl 6-(5-bromo-2-hydroxybenzoyl)-4-(4-chlorobutyl)-3-hydroxyphthalate (8q).



Starting with 6-brom-3-methoxalylchromone **(3b)** (0.309 g, 1.0 mmol) and 1-methoxy-8-chlor-1,3-bis(trimethylsilyloxy)-1,3-pentadiene (**5p**) (0.702 g, 2.0 mmol), Me_3SiOTf (0.36 mL, 2.0 mmol) in CH_2Cl_2 (4 mL), and then 5 h heating in EtOH (8 mL) with 3 mol% of p-TsOH, the product **8q** was isolated as a yellow oil (0.412 mg, 82%). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.72-1.88 (m, 4H, $(CH_2)_2$), 2.75 (t, ^3J = 7.0 Hz, 2H, CH_2), 3.57 (t, ^3J = 6.1 Hz, 2H, CH_2), 3.75 (s, 3H, OCH_3), 3.94 (s, 3H, OCH_3), 6.96 (d, ^3J = 8.7 Hz, 1H, Ar), 7.39 (bs, 1H, Ar), 7.48 (d, ^4J = 2.3 Hz, 1H, Ar), 7.58 (dd, ^3J = 8.8 Hz, ^4J = 2.5 Hz, 1H, Ar), 11.40 (s, 1H, OH), 11.55 (s, 1H, OH). ^{13}C NMR (75 MHz, $CDCl_3$): δ = 26.2, 28.9, 31.9, 44.6 (CH_2), 52.7, 53.4 (OCH_3), 110.3, 110.6 (C), 120.4 (Ar), 120.5, 127.1, 132.1,

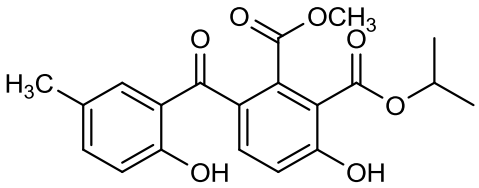
134.2 (C), 134.7, 135.0, 139.3 (Ar), 161.6, 161.9, 167.5, 169.3, 198.9 (C). IR (ATR, cm^{-1}): $\tilde{\nu}$ = 2951 (w), 1735 (s), 1675 (s), 1628 (s), 1602 (w), 1573 (w), 1462 (m), 1251 (s), 1189 (s), 1162 (s), 1052 (s), 808 (s), 692 (s). HRMS (ESI): calcd. for $C_{21}H_{20}BrClNaO_7$ ($[M+Na]^+$) 520.9973, found 520.9972; calcd. for $C_{21}H_{20}BrClNaO_7$ ($[M+Na]^+$) 522.9952, found 522.9955; calcd. for $C_{21}H_{20}BrClNaO_7$ ($[M+Na]^+$) 524.9935, found 524.9933. Anal. calcd. for $C_{21}H_{20}BrClO_7$ (499.74): C, 50.47; H, 4.03. Found: C, 50.48; H, 4.26.

Dimethyl 3-hydroxy-6-(2-hydroxy-5-methylbenzoyl)phthalate (8r).



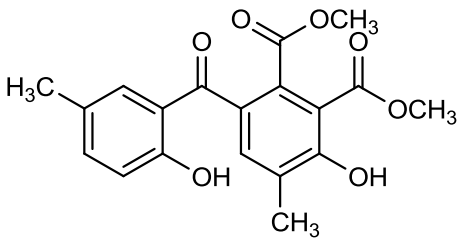
Starting with 6-methyl-3-methoxalylchromone **(3c)** (0.246 g, 1.0 mmol) and 1-methoxy-1,3-bis(trimethylsilyloxy)-1,3-butadiene (**5a**) (0.520 g, 2.0 mmol), Me_3SiOTf (0.36 mL, 2.0 mmol) in CH_2Cl_2 (4 mL), and then 5 h heating in EtOH (8 mL) with 3 mol% of p-TsOH, the product **8r** was isolated as a yellow solid (0.207 mg, 60%); mp = 116-120 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 2.22 (s, 3H, CH_3), 3.76 (s, 3H, OCH_3), 3.94 (s, 3H, OCH_3), 6.94 (d, ^3J = 8.4 Hz, 1H, Ar), 7.11-7.15 (m, 2H, Ar), 7.31 (dd, ^3J = 8.4 Hz, ^4J = 2.1 Hz, 1H, Ar), 7.56 (d, ^3J = 8.6 Hz, 1H, Ar), 11.09 (s, 1H, OH), 11.44 (s, 1H, OH). ^{13}C NMR (75 MHz, $CDCl_3$): δ = 20.3 (CH_3), 52.7, 53.3 (OCH_3), 110.9 (C), 118.1, 118.4 (Ar), 118.7, 128.0, 128.6 (C), 132.6, 135.3 (Ar), 136.3 (C), 137.8 (Ar), 160.9, 163.0, 167.6, 168.9, 199.5 (C). IR (ATR, cm^{-1}): $\tilde{\nu}$ = 3031 (w), 2953 (w), 1726 (s), 1674 (m), 1630 (m), 1612 (m), 1585 (m), 1440 (s), 1324 (s), 1210 (s), 1144 (s), 713 (s), 650 (s), 652 (s). GC-MS (EI, 70 eV): *m/z* (%) = 344 (M^+, 11), 312 (34), 253 (100), 252 (20). HRMS (EI, 70 eV): calcd. for $C_{18}H_{16}O_7$ (M^+) 344.08905, found 344.08950. Anal. calcd. for $C_{18}H_{16}O_7$ (344.32): C, 62.79; H, 4.68. Found: C, 62.77; H, 4.97.

2-Isopropyl 1-methyl 3-hydroxy-6-(2-hydroxy-5-methylbenzoyl)phthalate (8s).



Starting with 6-methyl-3-methoxalylchromone **(3c)** (0.246 g, 1.0 mmol) and 1-isopropoxy-1,3-bis(trimethylsilyloxy)-1,3-butadiene (**5d**) (0.577 g, 2.0 mmol), Me_3SiOTf (0.36 mL, 2.0 mmol) in CH_2Cl_2 (4 mL), and then 5 h heating in EtOH (8 mL) with 3 mol% of p-TsOH, the product **8s** was isolated as a yellow oil (0.236 mg, 63%).^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.34 (s, 3H, CH_3), 1.36 (s, 3H, CH_3), 2.22 (s, 3H, CH_3), 3.73 (s, 3H, OCH_3), 5.25-5.34 (m, 1H, CH), 6.94 (d, ^3J = 8.4 Hz, 1H, Ar), 7.11 (d, ^3J = 8.6 Hz, 1H, Ar), 7.15 (d, ^4J = 2.1 Hz, 1H, Ar), 7.31 (dd, ^3J = 8.4 Hz, ^4J = 2.1 Hz, 1H, Ar), 7.54 (d, ^3J = 8.7 Hz, 1H, Ar), 11.34 (s, 1H, OH), 11.47 (s, 1H, OH). ^{13}C NMR (75 MHz, $CDCl_3$): δ = 20.4, 21.4 (CH_3), 52.5 (OCH_3), 71.2 (CH), 111.2 (C), 118.1, 118.3 (Ar), 118.8, 128.0, 128.4 (C), 132.7, 135.0 (Ar), 136.2 (C), 137.8 (Ar), 160.9, 163.3, 167.4, 168.2, 199.7 (C). IR (ATR, cm^{-1}): $\tilde{\nu}$ = 2984 (w), 1736 (m), 1671 (m), 1631 (m), 1608 (m), 1582 (m), 1320 (s), 1215 (s), 1096 (s), 802 (s), 647 (s). GC-MS (EI, 70 eV): *m/z* (%) = 372 (M^+, 14), 340 (39), 298 (74), 253 (100). HRMS (ESI): calcd. for $C_{20}H_{20}NaO_7$ ($[M+Na]^+$) 395.1101, found 395.1100.

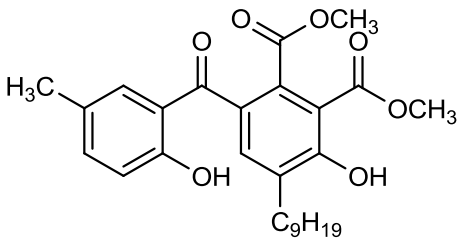
Dimethyl 3-hydroxy-6-(2-hydroxy-5-methylbenzoyl)-4-methylphthalate (8t).



Starting with 6-methyl-3-methoxalylchromone **(3c)** (0.246 g, 1.0 mmol) and 1-methoxy-1,3-bis(trimethylsilyloxy)-1,3-pentadiene (**5i**) (0.549 g, 2.0 mmol), Me_3SiOTf (0.36 mL, 2.0 mmol) in CH_2Cl_2 (4 mL), and then 5 h heating in EtOH (8 mL) with 3 mol% of p-TsOH, the product **8t** was isolated as a yellow solid (0.264 mg, 74%); mp = 110-114 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 2.22 (s, 3H, CH_3), 2.32 (s, 3H, CH_3), 3.71 (s, 3H, OCH_3), 3.93 (s, 3H, OCH_3), 6.94 (d, ^3J = 8.4 Hz, 1H, Ar), 7.14 (d, ^4J = 2.1 Hz, 1H, Ar), 7.31 (dd, ^3J = 8.4 Hz, ^4J = 2.2 Hz, 1H, Ar), 7.40 (bs, 1H, Ar), 11.28 (s, 1H, OH), 11.47 (s, 1H, OH). ^{13}C NMR (75 MHz, $CDCl_3$): δ = 16.0, 20.3 (CH_3), 52.6, 53.2 (OCH_3), 110.1 (C), 118.0 (Ar), 118.9, 127.9, 128.1, 128.4, (C), 132.7 (Ar), 133.6 (C), 135.2, 137.7 (Ar), 160.8, 161.4, 167.7, 169.5, 200.0 (C). IR (ATR, cm^{-1}): $\tilde{\nu}$ = 3030 (w), 2995 (w), 2950 (w), 2923 (w), 1736 (s), 1686 (s), 1630 (s), 1598 (s), 1484 (s), 1438 (s), 1351 (s), 1242 (s), 1206 (s), 1155 (s), 1049 (s), 986 (s), 815 (s), 671 (s). GC-MS (EI, 70 eV): *m/z* (%) = 358 (M^+, 10), 326 (36), 294

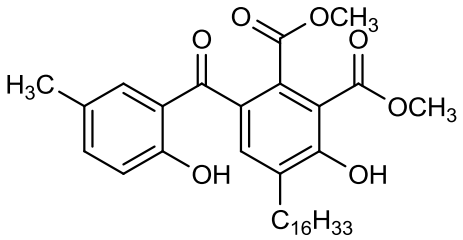
(35), 267 (100). HRMS (EI, 70 eV): calcd. for $C_{19}H_{18}O_7$ (M^+) 358.10470, found 358.10607. Anal. calcd. for $C_{19}H_{18}O_7$ (358.34): C, 63.68; H, 5.06. Found: C, 63.66; H, 5.20.

Dimethyl 3-hydroxy-6-(2-hydroxy-5-methylbenzoyl)-4-nonylphthalate (8v).



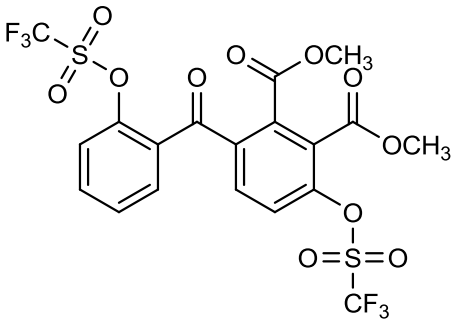
Starting with 6-methyl-3-methoxalylchromone **(3c)** (0.246 g, 1.0 mmol) and 1-methoxy-1,3-bis(trimethylsilyloxy)-1,3-tridecadiene (**5k**) (0.773 g, 2.0 mmol), Me_3SiOTf (0.36 mL, 2.0 mmol) in CH_2Cl_2 (4 mL), and then 5 h heating in EtOH (8 mL) with 3 mol% of p-TsOH, the product **8v** was isolated as a yellow oil (0.298 mg, 63%). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.86 (t, ^3J = 6.7 Hz, 3H, CH_3), 1.20-1.36 (m, 12H, $(CH_2)_6$), 1.55-1.65 (m, 2H, CH_2), 2.22 (s, 3H, CH_3), 2.69 (t, ^3J = 7.5 Hz, 2H, CH_2), 3.74 (s, 3H, OCH_3), 3.93 (s, 3H, OCH_3), 6.95 (d, ^3J = 8.4 Hz, 1H, Ar), 7.16 (d, ^4J = 2.0 Hz, 1H, Ar), 7.32 (dd, ^3J = 8.5 Hz, ^4J = 2.2 Hz, 1H, Ar), 7.40 (bs, 1H, Ar), 11.31 (s, 1H, OH), 11.50 (s, 1H, OH). ^{13}C NMR (75 MHz, $CDCl_3$): δ = 14.0, 20.3 (CH_3), 22.6, 28.9, 29.2, 29.3, 29.4, 29.5, 29.7, 31.8 (CH_2), 52.6, 53.2 (OCH_3), 110.3 (C), 118.1 (Ar), 118.9, 127.9, 128.0, 132.7 (C), 132.8 (Ar), 133.7 (C), 134.9, 137.7 (Ar), 160.9, 161.2, 167.9, 169.5, 200.0 (C). IR (ATR, cm^{-1}): $\tilde{\nu}$ = 2923 (s), 2853 (m), 1737 (s), 1675 (s), 1632 (s), 1607 (m), 1482 (m), 1436 (s), 1354 (s), 1251 (s), 1206 (s), 1153 (s), 1052 (s), 719 (s), 675 (s). MS (EI, 70 eV): *m/z* (%) = 470 (M^+, 8), 438 (70), 406 (29), 379 (100), 294 (42). HRMS (ESI): calcd. for $C_{27}H_{35}O_7$ ($[M+H]^+$) 471.2383, found 471.2383; calcd. for $C_{27}H_{34}NaO_7$ ($[M+Na]^+$) 493.2196, found 493.2203. Anal. calcd. for $C_{27}H_{34}O_7$ (470.55): C, 68.92; H, 7.28. Found: C, 69.06; H, 7.22.

Dimethyl 4-hexadecyl-3-hydroxy-6-(2-hydroxy-5-methylbenzoyl)phthalate (8w).



Starting with 6-methyl-3-methoxalylchromone **(3c)** (0.246 g, 1.0 mmol) and 1-methoxy-1,3-bis(trimethylsilyloxy)-1,3-icosadiene (**5m**) (0.969 g, 2.0 mmol), Me_3SiOTf (0.36 mL, 2.0 mmol) in CH_2Cl_2 (4 mL), and then 5 h heating in EtOH (8 mL) with 3 mol% of p-TsOH, the product **8w** was isolated as a orange solid (0.409 mg, 72%); mp = 65-68 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.87 (t, ^3J = 6.6 Hz, 3H, CH_3), 1.24-1.30 (m, 26H, $(CH_2)_{13}$), 1.55-1.62 (m, 2H, CH_2), 2.22 (s, 2H, CH_3), 2.69 (t, ^3J = 7.5 Hz, 2H, CH_2), 3.74 (s, 3H, OCH_3), 3.93 (s, 3H, OCH_3), 6.95 (d, ^3J = 8.4 Hz, 1H, Ar), 7.16 (d, ^4J = 1.8 Hz, 1H, Ar), 7.31 (dd, ^3J = 8.5 Hz, ^4J = 2.1 Hz, 1H, Ar), 7.39 (bs, 1H, Ar), 11.31 (s, 1H, OH), 11.50 (s, 1H, OH). ^{13}C NMR (75 MHz, $CDCl_3$): δ = 14.1, 20.3 (CH_3), 22.6, 28.9, 29.3, 29.4, 29.5, 29.6, 29.7, 29.8, 31.9 (CH_2), 52.6, 53.2 (OCH_3), 110.3 (C), 118.1 (Ar), 118.9, 127.9, 128.0, 132.7 (C), 132.9 (Ar), 133.7 (C), 134.9, 137.7 (Ar), 160.9, 161.2, 167.8, 169.5, 200.0 (C). IR (ATR, cm^{-1}): $\tilde{\nu}$ = 2954 (w), 2919 (s), 2852 (s), 1733 (s), 1672 (s), 1632 (s), 1583 (w), 1488 (w), 1437 (s), 1207 (s), 1154 (s), 981 (s), 708 (s). MS (EI, 70 eV): *m/z* (%) = 568 (M^+, 5), 537 (41), 536 (100), 504 (25), 477 (44), 294 (28). HRMS (ESI): calcd. for $C_{34}H_{49}O_7$ ($[M+H]^+$) 569.3472, found 569.3471; calcd. for $C_{34}H_{48}NaO_7$ ($[M+Na]^+$) 591.3292, found 591.3301. Anal. calcd. for $C_{34}H_{48}O_7$ (568.74): C, 71.80; H, 8.51. Found: C, 71.78; H, 8.82.

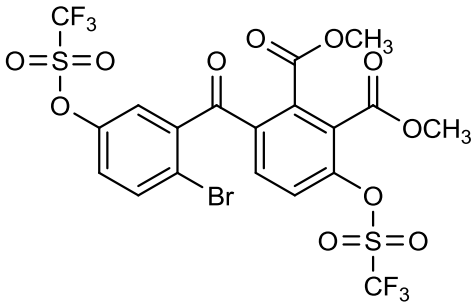
Dimethyl 3-(trifluoromethylsulfonyloxy)-6-(2-(trifluoromethylsulfonyloxy)benzoyl) phthalate (9a).



Starting with dimethyl 3-hydroxy-6-(2-hydroxybenzoyl)phthalate (**8a**) (0.642 g, 1.9 mmol), pyridine (0.6 ml, 7.6 mmol) and Tf_2O (0.7 ml, 4.6 mmol) in CH_2Cl_2 (19 mL), the product **9a** was isolated as a yellow oil (0.738 g, 64%). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 3.74 (s, 3H, OCH_3), 4.01 (s, 3H, OCH_3), 7.64-7.77 (m, 6H, Ar). ^{13}C NMR (75 MHz, $CDCl_3$): δ = 53.3, 53.4 (OCH_3), 118.5, 118.6 (q, $J_{C\text{-}F}$ = 319.6 Hz, CF_3), 122.9, 123.9 (Ar), 127.6 (C), 128.4 (Ar), 130.2 (C), 132.6, 134.6 (Ar), 138.5, 147.3, 147.9, 163.2, 165.1, 190.5 (C). ^{19}F NMR (235 MHz, $CDCl_3$): δ = -72.9, -72.8 (CF_3). IR (ATR, cm^{-1}): $\tilde{\nu}$ = 2954 (w), 1733 (s), 1672 (s), 1630 (s), 1594 (m), 1464 (m), 1447 (m), 1255 (s), 1157 (s), 1053 (s), 802 (s), 683 (s). HRMS (ESI): calcd. for $C_{19}H_{13}F_6O_{11}S_2$

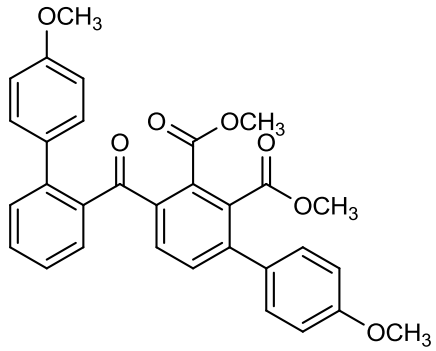
([M+H]$^+$) 594.9798, found 594.9802; calcd. for $C_{19}H_{12}F_6NaO_{11}S_2$ ([M+Na]$^+$) 616.9617, found 616.9616. Anal. calcd. for $C_{19}H_{12}F_6O_{11}S_2$ (594.41): C, 38.39; H, 2.03; S, 10.79. Found: C, 39.09; H, 2.21; S, 11.01.

Dimethyl 3-(2-bromo-5-(trifluoromethylsulfonyloxy)benzoyl)-6-(trifluoromethylsulfonyloxy)phthalate (9b).



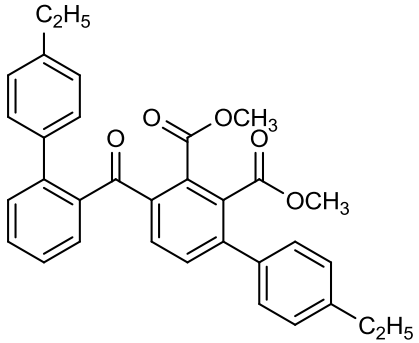
Starting with dimethyl 3-(5-bromo-2-hydroxybenzoyl)-6-hydroxyphthalate (**8l**) (1.0 g, 2.4 mmol), pyridine (0.7 ml, 9.7 mmol) and Tf_2O (0.9 ml, 5.8 mmol) in CH_2Cl_2 (25 mL), the product **9b** was isolated as a yellow solid (1.2 g, 73%); mp = 86-87 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 3.73 (s, 3H, OCH_3), 3.97 (s, 3H, OCH_3), 7.29 (d, ^3J = 8.7 Hz, 1H, Ar), 7.54 (d, ^3J = 8.6 Hz, 1H, Ar), 7.65-7.69 (m, 2H, Ar), 7.80 (dd, ^3J = 8.7 Hz, ^4J = 2.4 Hz, 1H, Ar). ^{13}C NMR (75 MHz, $CDCl_3$): δ = 53.3, 53.4 (OCH_3), 118.4, 118.5 (q, $J_{C\text{-}F}$ = 320.6 Hz, CF_3), 122.0 (C), 124.0, 124.4 (Ar), 127.7, 131.8 (C), 132.6, 134.8, 137.3 (Ar), 137.6, 146.0, 148.2, 163.0, 165.0, 189.2 (C). ^{19}F NMR (235 MHz, $CDCl_3$): δ = -72.8, -72.6 (CF_3). IR (ATR, cm^{-1}): $\tilde{\nu}$ = 3096 (w), 2952 (w), 1736 (s), 1696 (m), 1584 (w), 1422 (s), 1385 (w), 1207 (s), 1135 (s), 1000 (s), 824 (s), 605 (s). HRMS (ESI): calcd. for $C_{19}H_{12}Br^{79}F_6O_{11}S_2$ ([M+H]$^+$) 672.8903, found 672.8891; calcd. for $C_{19}H_{12}Br^{81}F_6O_{11}S_2$ ([M+H]$^+$) 674.8884, found 674.8877; calcd. for $C_{19}H_{11}Br^{79}F_6NaO_{11}S_2$ ([M+Na]$^+$) 694.8722, found 694.8727; calcd. for $C_{19}H_{11}Br^{81}F_6NaO_{11}S_2$ ([M+Na]$^+$) 696.8703, found 696.8709. Anal. calcd. for $C_{19}H_{11}BrF_6O_{11}S_2$ (673.31): C, 33.89; H, 1.65; S, 9.52. Found: C, 34.38; H, 1.77; S, 10.37.

Dimethyl 4'-methoxy-4-(4'-methoxybiphenylcarbonyl)biphenyl-2,3-dicarboxylate (10a).



Starting with dimethyl 3-(trifluoromethylsulfonyloxy)-6-(2-(trifluoromethylsulfonyloxy) benzoyl) phthalate (**9a**) (0.382 g, 0.7 mmol), K_3PO_4 (0.408 g, 1.9 mmol), $Pd(PPh_3)_4$ (6mol%) and 4-methoxyphenylboronic acid (0.244 g, 1.6 mmol) in 1,4-dioxane (3 mL), the product (**10a**) was isolated as a yellow solid (0.195 g, 60%); mp = 59-62 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 3.61 (s, 3H, OCH_3), 3.72 (s, 3H, OCH_3), 3.75 (s, 3H, OCH_3), 3.83 (s, 3H, OCH_3), 6.74-7.68 (m, 14H, Ar). ^{13}C NMR (75 MHz, $CDCl_3$): δ = 52.4, 52.7, 55.2, 55.3 (OCH_3), 113.7, 113.9, 126.8, 129.2, 130.3, 130.4, 130.5, 130.8 (Ar), 131.3 (C), 131.5 (Ar), 132.0, 132.2, 136.4, 137.3, 142.0, 143.2, 159.0, 159.6, 167.9, 168.1, 196.8 (C). IR (ATR, cm^{-1}): $\tilde{\nu}$ = 2948 (w), 2836 (w), 1726 (s), 1659 (m), 1607 (m), 1579 (m), 1514 (s), 1240 (s), 1177 (s), 829 (s), 762 (s). GC-MS (EI, 70 eV): *m/z* (%) = 510 (M^+, 100), 419 (43), 211 (31). HRMS (ESI): calcd. for $C_{31}H_{27}O_7$ ($[M+H]^+$) 511.1751, found 511.1757; calcd. for $C_{31}H_{26}NaO_7$ ($[M+Na]^+$) 533.1570, found 533.1582. Anal. calcd. for $C_{31}H_{26}O_{11}$ (510.53): C, 72.93; H, 5.13. Found: C, 72.78; H, 5.56.

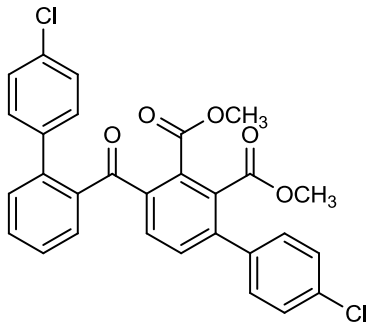
Dimethyl 4'-ethyl-4-(4'-ethylbiphenylcarbonyl)biphenyl-2,3-dicarboxylate (10b).



Starting with dimethyl 3-(trifluoromethylsulfonyloxy)-6-(2-(trifluoromethylsulfonyloxy) benzoyl) phthalate (**9a**) (0.356 g, 0.6 mmol), K_3PO_4 (0.382 g, 1.8 mmol), $Pd(PPh_3)_4$ (6mol%) and 4-ethylphenylboronic acid (0.224 g, 1.5 mmol) in 1,4-dioxane (3 mL), the product (**10b**) was isolated as a white solid (0.183 g, 60%); mp = 48-50 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.21 (t, ^3J = 7.5 Hz, 3H, CH_3), 1.30 (t, ^3J = 7.5 Hz, 3H, CH_3), 2.61 (q, ^3J = 7.5 Hz, 2H, CH_2), 2.73 (q, ^3J = 7.5 Hz, 2H, CH_2), 3.64 (s, 3H, OCH_3), 3.79 (s, 3H, OCH_3), 7.09-7.74 (m, 15H, Ar + $CHCl_3$). ^{13}C NMR (75 MHz, $CDCl_3$): δ = 15.3, 15.5 (CH_3), 28.4, 28.5 (CH_2), 52.3, 52.7 (OCH_3), 126.9, 127.7, 127.9, 128.0, 129.2, 130.3, 130.4, 130.7, 131.4, 131.5 (Ar), 132.0, 132.6, 136.3, 136.5, 137.1, 137.4, 142.4, 143.5, 144.3, 167.9, 168.0, 196.8 (C). IR (ATR, cm^{-1}): $\tilde{\nu}$ = 2962 (w), 2871 (w), 1728 (s), 1661 (m), 1612 (w), 1585 (m), 1515 (w), 1232 (s), 1150 (s), 940 (s), 828 (s), 761 (s). GC-MS (EI, 70 eV): *m/z*

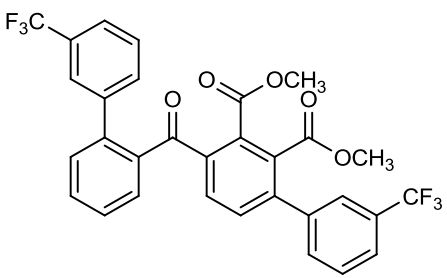
(%) = 506 (M^+, 5), 474 (43), 443 (42), 442 (100), 424 (52), 414 (45), 413 (67), 207 (41). HRMS (ESI): calcd. for $C_{33}H_{31}O_5$ ($[M+H]^+$) 507.2166, found 507.2178; calcd. for $C_{31}H_{30}NaO_5$ ($[M+Na]^+$) 529.1985, found 529.1998. Anal. calcd. for $C_{33}H_{30}O_5$ (506.59): C, 78.24; H, 5.97. Found: C, 78.24; H, 6.16.

Dimethyl 4'-chloro-4-(4'-chlorobiphenylcarbonyl)biphenyl-2,3-dicarboxylate (10c).



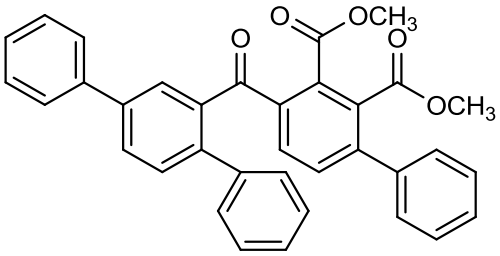
Starting with dimethyl 3-(trifluoromethylsulfonyloxy)-6-(2-(trifluoromethylsulfonyloxy) benzoyl) phthalate (**9a**) (0.356 g, 0.6 mmol), K_3PO_4 (0.382 g, 1.8 mmol), $Pd(PPh_3)_4$ (6mol%) and 4-chlorophenylboronic acid (0.234 g, 1.5 mmol) in 1,4-dioxane (3 mL), the product (**10c**) was isolated as a yellow solid (0.218 g, 70%); mp = 148-150 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 3.53 (s, 3H, OCH_3), 3.58 (s, 3H, OCH_3), 7.10-7.65 (m, 15H, Ar + $CHCl_3$). ^{13}C NMR (75 MHz, $CDCl_3$): δ = 52.6, 52.9 (OCH_3), 127.5, 128.2, 128.7, 129.3, 130.5, 130.6, 131.0, 131.3, 131.8 (Ar), 132.2, 132.3, 133.5, 134.5, 137.1, 137.3, 137.4, 138.3, 141.2, 142.4, 167.3, 167.5, 196.3 (C). IR (ATR, cm^{-1}): $\tilde{\nu}$ = 2955 (w), 1938 (w), 1747 (s), 1722 (s), 1663 (m), 1583 (w), 1494 (w), 1253 (s), 1151 (s), 1086 (s), 828 (s), 785 (s), 674 (s). GC-MS (EI, 70 eV): *m/z* (%) = 520 (M^+, 39), 519 (M^+, 24), 518 (M^+, 59), 429 (65), 428 (26), 427 (100), 333 (24), 332 (11), 331 (73), 215 (62), 152 (51). HRMS (ESI): calcd. for $C_{29}H_{20}Cl_2NaO_5$ ($[M+Na]^+$) 541.0580, found 541.0578; calcd. for $C_{29}H_{20}Cl_2NaO_5$ ($[M+Na]^+$) 543.0558, found 543.0569. Anal. calcd. for $C_{29}H_{20}Cl_2O_5$ (519.37): C, 67.06; H, 3.88. Found: C, 66.96; H, 3.88.

Dimethyl 3'-(trifluoromethyl)-4-(3'-(trifluoromethyl)biphenylcarbonyl)biphenyl-2,3-dicarboxylate (10d).



Starting with dimethyl 3-(trifluoromethylsulfonyloxy)-6-(2-(trifluoromethylsulfonyloxy) benzoyl) phthalate (**9a**) (0.356 g, 0.6 mmol), K_3PO_4 (0.382 g, 1.8 mmol), $Pd(PPh_3)_4$ (6mol%) and 3-(trifluoromethyl)phenylboronic acid (0.284 g, 1.5 mmol) in 1,4-dioxane (3 mL), the product (**10d**) was isolated as a yellow oil (0.137 g, 39%). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 3.63 (s, 3H, OCH_3), 3.68 (s, 3H, OCH_3), 7.29-7.82 (m, 16H, Ar + $CHCl_3$). ^{13}C NMR (75 MHz, $CDCl_3$): δ = 52.4, 52.8 (OCH_3), 124.1, 124.8, 125.0, 125.7 (q, $J_{C\text{-}F}$ = 3.9 Hz, CH), 127.9, 128.7, 129.0 (Ar), 129.5, 130.1, 130.6, 137 (C), 130.8, 131.1, 131.2, 131.4, 132.0 (Ar), 132.3, 132.5 (C), 132.8 (Ar), 137.0, 137.8, 139.5, 140.7, 141.0, 141.9, 167.1, 167.3, 196.0 (C). ^{19}F NMR (235 MHz, $CDCl_3$): δ = -62.3, -62.2 (CF_3). IR (ATR, cm^{-1}): $\tilde{\nu}$ = 3011 (w), 2957 (w), 1726 (s), 1661 (m), 1592 (w), 1428 (w), 1406 (w), 1333 (s), 1239 (s), 1116 (s), 1067 (s), 808 (s), 767 (s), 695 (s). GC-MS (EI, 70 eV): *m/z* (%) = 586 (M^+, 27), 495 (61), 365 (100), 249 (71), 201 (35). HRMS (ESI): calcd. for $C_{31}H_{20}F_6NaO_5$ ($[M+Na]^+$) 609.1107, found 609.1100. Anal. calcd. for $C_{31}H_{20}F_6O_5$ (586.48): C, 63.40; H, 3.44. Found: C, 63.46; H, 3.57.

Dimethyl 4-(4-phenylbiphenylcarbonyl)biphenyl-2,3-dicarboxylate (11).

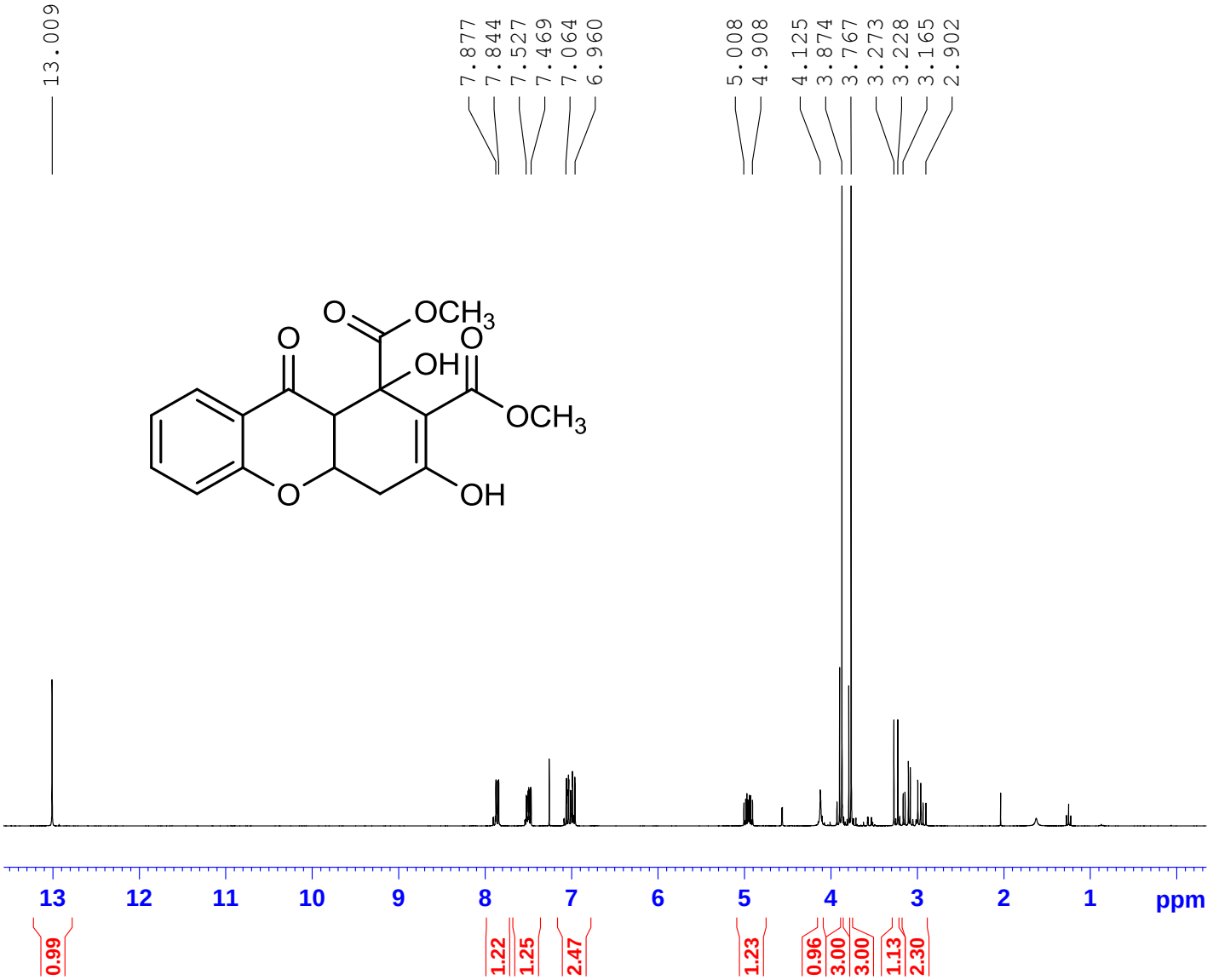


Starting with (**9b**) (0.336 g, 0.5 mmol), KF (0.130 g, 2.2 mmol), $Pd(PPh_3)_4$ (6mol%) and phenylboronic acid (0.243 g, 2.0 mmol) in 1,4-dioxane (2.5 mL), the product (**11**) was isolated as a yellow solid (0.130 g, 50%); mp = 155-157 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 3.49 (s, 3H, OCH_3), 3.64 (s, 3H, OCH_3), 7.10-7.83 (m, 23H, Ar + $CHCl_3$). ^{13}C NMR (75 MHz, $CDCl_3$): δ = 52.4, 52.8 (OCH_3), 127.0, 124.4, 127.9, 128.2, 128.3, 128.4, 128.8, 129.0, 129.2 (Ar), 130.0, 130.9, 131.1, 131.6, 132.8, 136.6, 137.8, 139.0, 139.4, 140.1, 141.2, 143.7, 167.8, 196.6 (C). IR (ATR, cm^{-1}): $\tilde{\nu}$ = 3050 (w), 3028 (w),

2996 (w), 2948 (w), 2855 (w), 1739 (s), 1723 (s), 1588 (w), 1473 (w), 1232 (s), 1150 (s), 754 (s), 692 (s). GC-MS (EI, 70 eV): *m/z* (%) = 526 (M^+, 88), 525 (39), 436 (42), 435 (100), 297 (45), 257 (44), 228 (33). HRMS (ESI): calcd. for $C_{35}H_{26}NaO_5$ ($[M+Na]^+$) 549.1672, found 549.1670. Anal. calcd. for $C_{35}H_{26}O_5$ (526.58): C, 79.83; H, 4.98. Found: C, 79.90; H, 4.91.

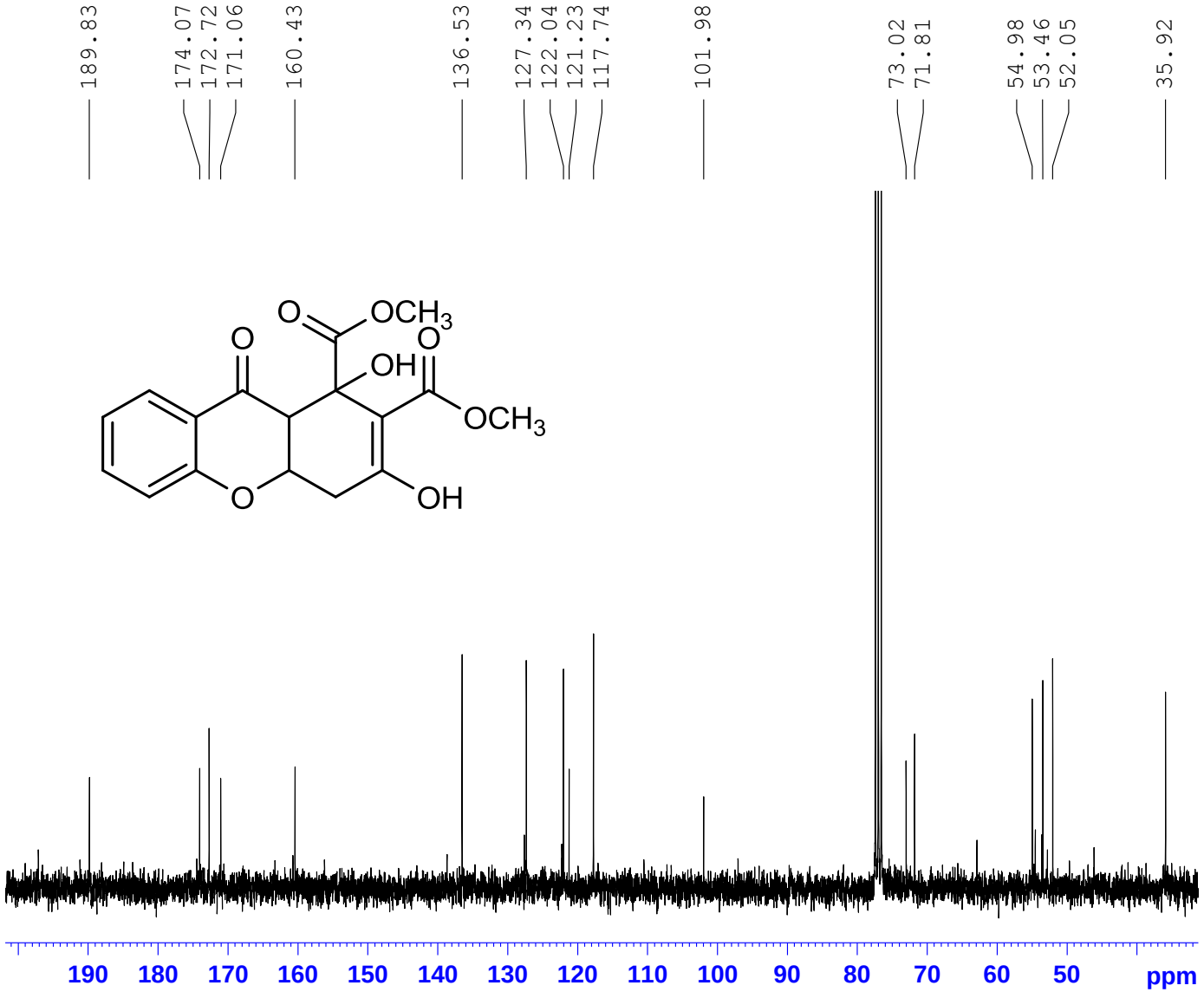
(C) Copies of ^{1}H and ^{13}C NMR spectra

Bunescu, AB287
Au1H CDCl3 /opt/topspin 1005 22




```
Current Data Parameters
NAME            AB287 1H
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20100526
Time               23.55
INSTRUM            AV300
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     4
SWH             6172.839 Hz
FIDRES          0.188380 Hz
AQ              2.6542580 sec
RG                 322.5
DW                81.000 usec
DE                 6.00 usec
TE                295.4 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.00 usec
PL1                 0.00 dB
SFO1         300.1318534 MHz

F2 - Processing parameters
SI                 32768
SF           300.1300153 MHz
WDW                   EM
SSB                    0
LB                  0.10 Hz
GB                     0
PC                  1.00
```

Bunescu, AB287
Au13C CDCl3 /opt/topspin 1005 22



Current Data Parameters
NAME AB287 13C
EXPNO 11
PROCNO 1

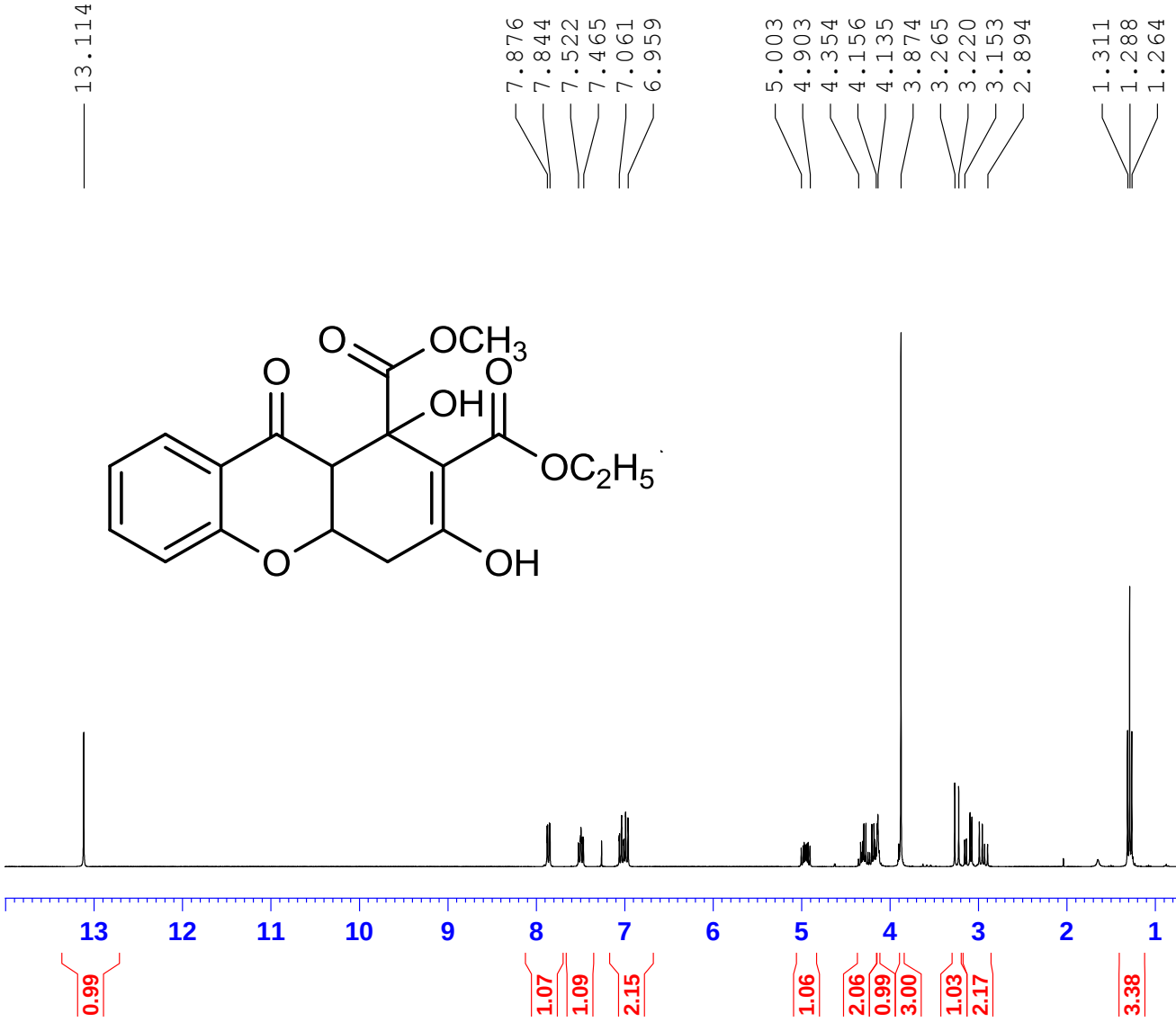
F2 - Acquisition Parameters
Date_ 20100527
Time 0.08
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 256
DS 4
SWH 21097.047 Hz
FIDRES 0.643831 Hz
AQ 0.7766516 sec
RG 32768
DW 23.700 usec
DE 6.00 usec
TE 295.7 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.60 usec
PL1 -1.10 dB
SFO1 75.4771825 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 19.00 dB
PL13 21.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677539 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Bunescu, AB 272
Au1H CDCl3 /opt/topspin 1004 29



Current Data Parameters
NAME AB272 1H
EXPNO 10
PROCNO 1

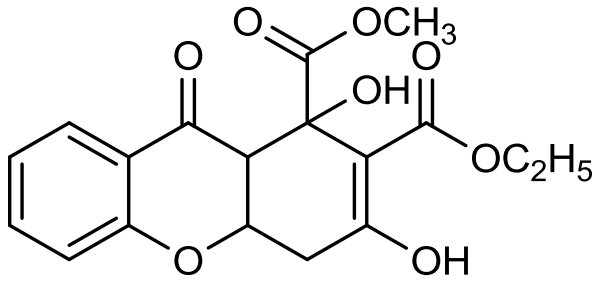
F2 - Acquisition Parameters
Date_ 20100501
Time 3.44
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 4
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 322.5
DW 81.000 usec
DE 6.00 usec
TE 294.8 K
D1 1.00000000 sec
TD0 1

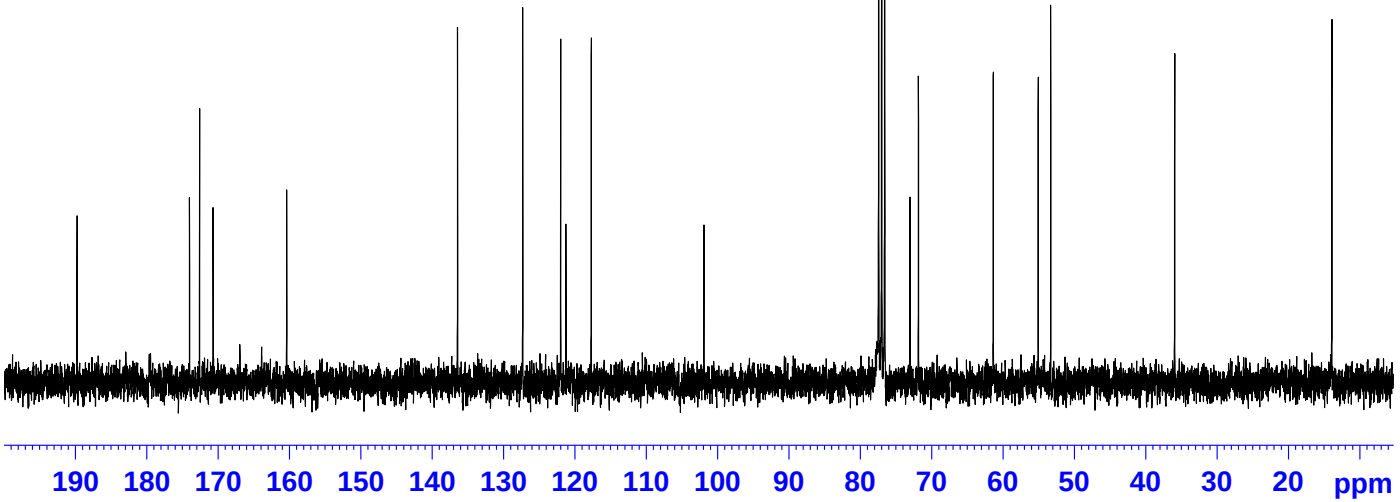
======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 0.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300153 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

Bunescu, AB 272
Au13C CDCl3 /opt/topspin 1004 29

189.76 174.00 172.58 170.69 160.42 136.46 127.33 122.00 121.28 117.72 101.92 73.04 71.85 61.36 55.07 53.32 35.94 13.90





Current Data Parameters
NAME AB272 13C
EXPNO 11
PROCNO 1

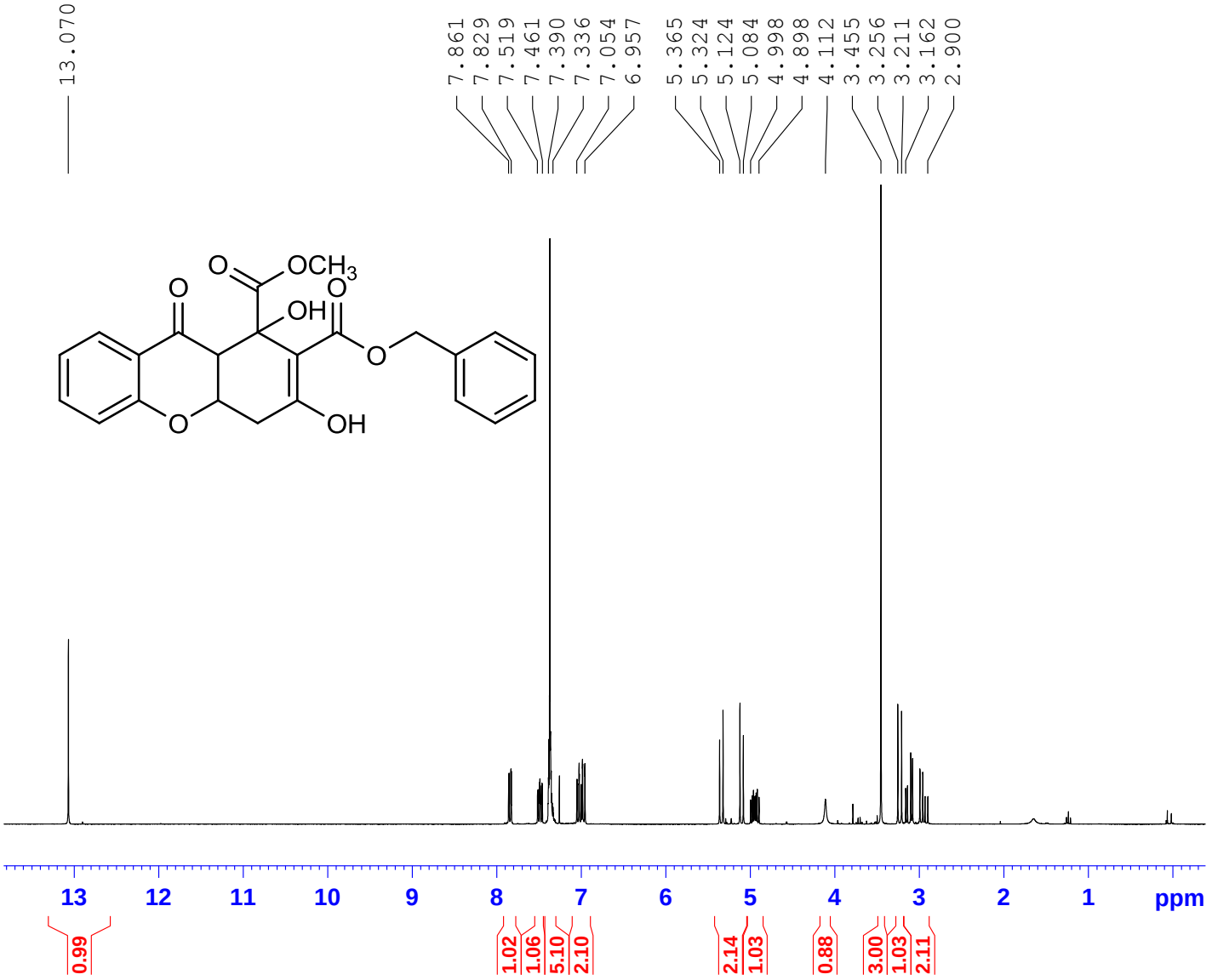
F2 - Acquisition Parameters
Date_ 20100501
Time 3.57
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 256
DS 4
SWH 21097.047 Hz
FIDRES 0.643831 Hz
AQ 0.7766516 sec
RG 32768
DW 23.700 usec
DE 6.00 usec
TE 295.4 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.60 usec
PL1 -1.10 dB
SFO1 75.4771825 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 19.00 dB
PL13 21.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677547 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Bunescu/ AB 294
Au1H CDCl3 /opt/topspin 1006 20



Current Data Parameters
NAME AB294 1H
EXPNO 10
PROCNO 1

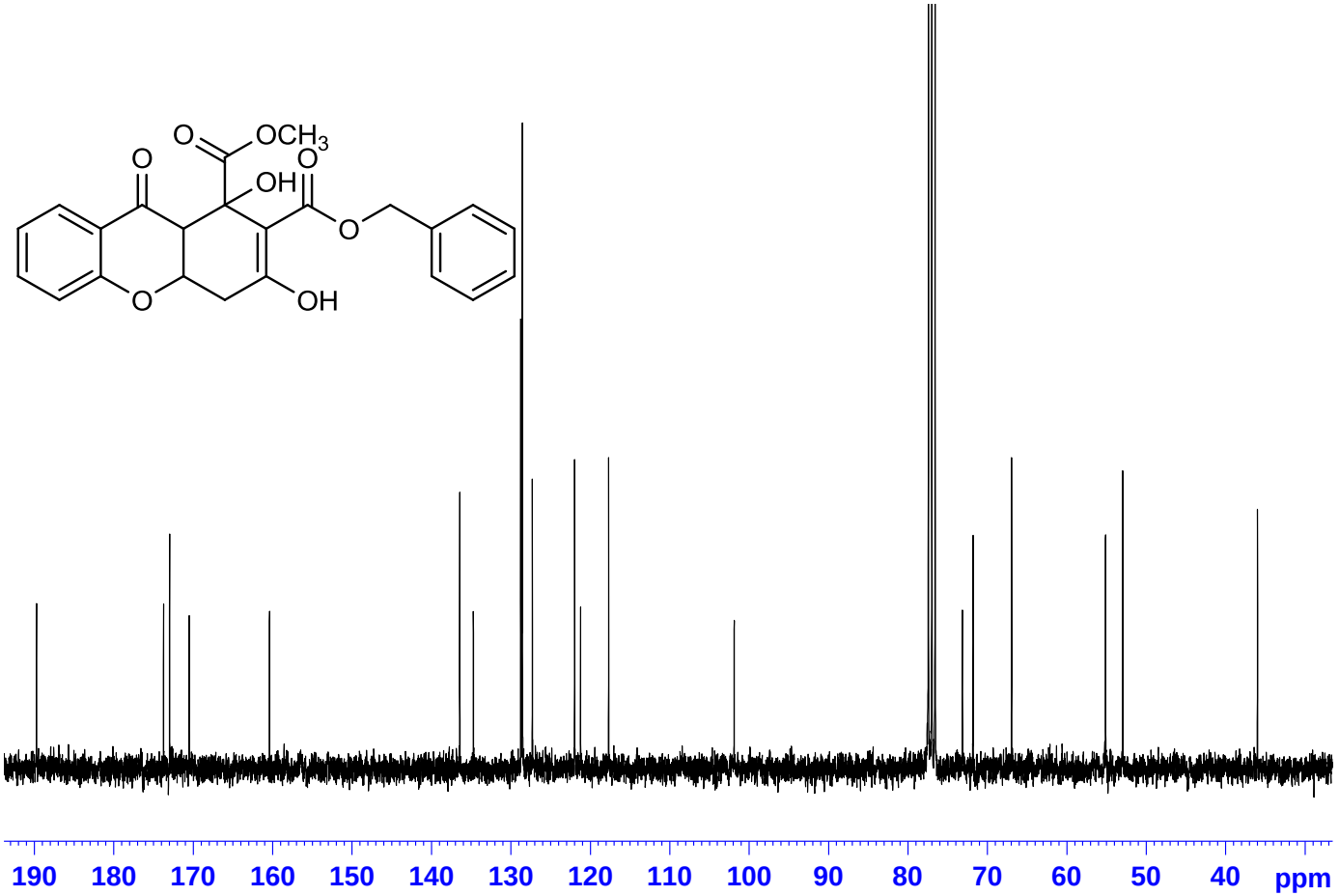
F2 - Acquisition Parameters
Date_ 20100626
Time 8.53
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 4
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 322.5
DW 81.000 usec
DE 6.00 usec
TE 295.6 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 0.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300153 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

Bunescu/ AB 294
Au13C CDCl3 /opt/topspin 1006 20

189.73 173.72 172.96 170.50 160.38 136.45 134.73 128.77 128.62 128.57 127.31 121.99 121.24 117.71 101.89 73.15 71.80 66.96 55.13 52.97 35.98



Current Data Parameters
NAME AB294 13C
EXPNO 11
PROCNO 1

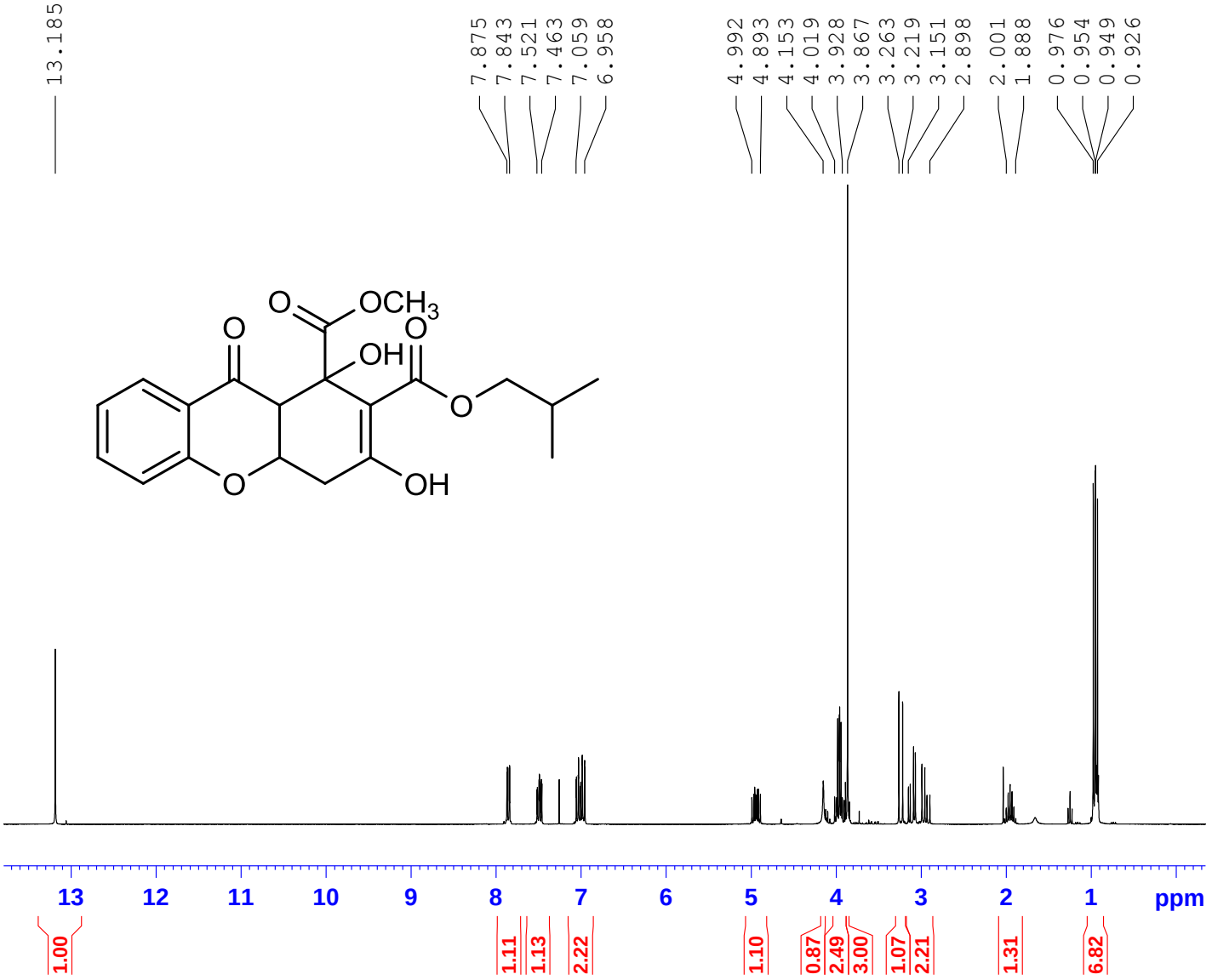
F2 - Acquisition Parameters
Date_ 20100626
Time 9.08
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 256
DS 4
SWH 21097.047 Hz
FIDRES 0.643831 Hz
AQ 0.7766516 sec
RG 32768
DW 23.700 usec
DE 6.00 usec
TE 296.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.60 usec
PL1 -1.10 dB
SFO1 75.4771825 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 19.00 dB
PL13 21.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677553 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Bunescu/ AB 295
Au1H CDCl3 /opt/topspin 1006 21



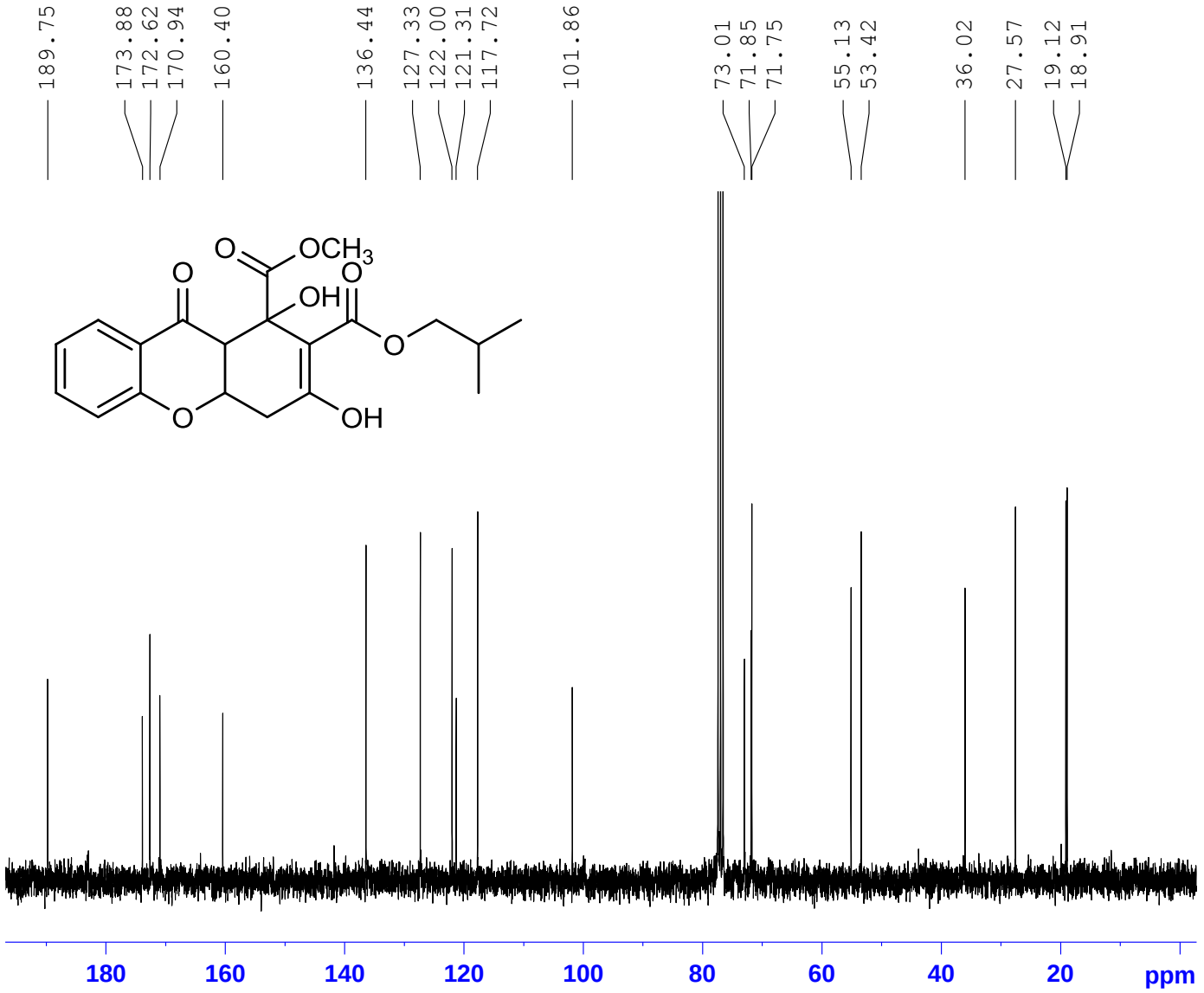
Current Data Parameters
NAME AB295 1H
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100626
Time 9.26
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 4
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 161.3
DW 81.000 usec
DE 6.00 usec
TE 295.6 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 0.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300154 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

Bunescu/ AB 295
Au13C CDCl3 /opt/topspin 1006 21



Current Data Parameters
NAME AB295 13C
EXPNO 11
PROCNO 1

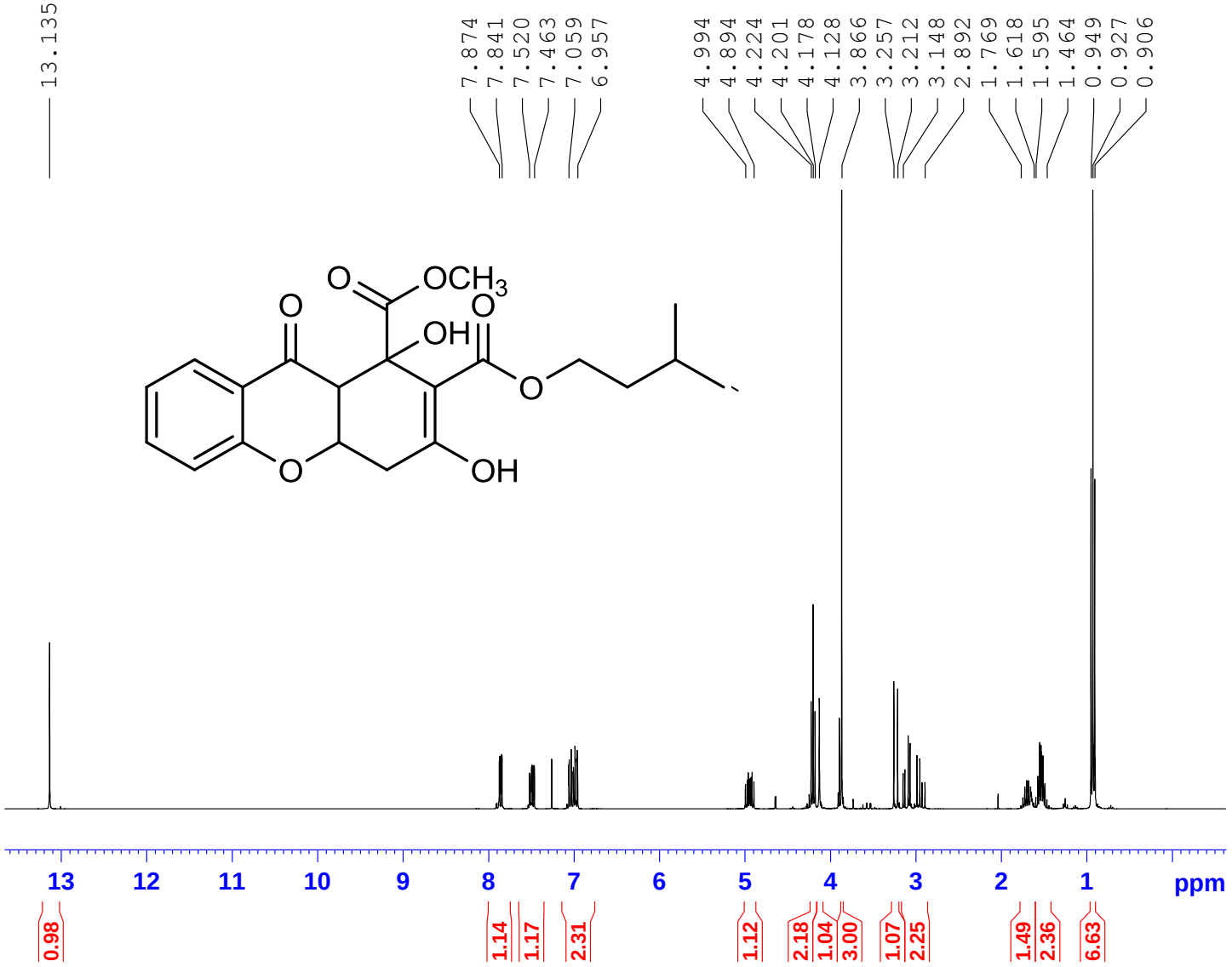
F2 - Acquisition Parameters
Date_ 20100626
Time 9.39
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 256
DS 4
SWH 21097.047 Hz
FIDRES 0.643831 Hz
AQ 0.7766516 sec
RG 32768
DW 23.700 usec
DE 6.00 usec
TE 295.9 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.60 usec
PL1 -1.10 dB
SFO1 75.4771825 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 19.00 dB
PL13 21.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677545 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Bunescu/ AB 290
Au1H CDCl3 /opt/topspin 1006 12



Current Data Parameters
NAME AB290 1H
EXPNO 10
PROCNO 1

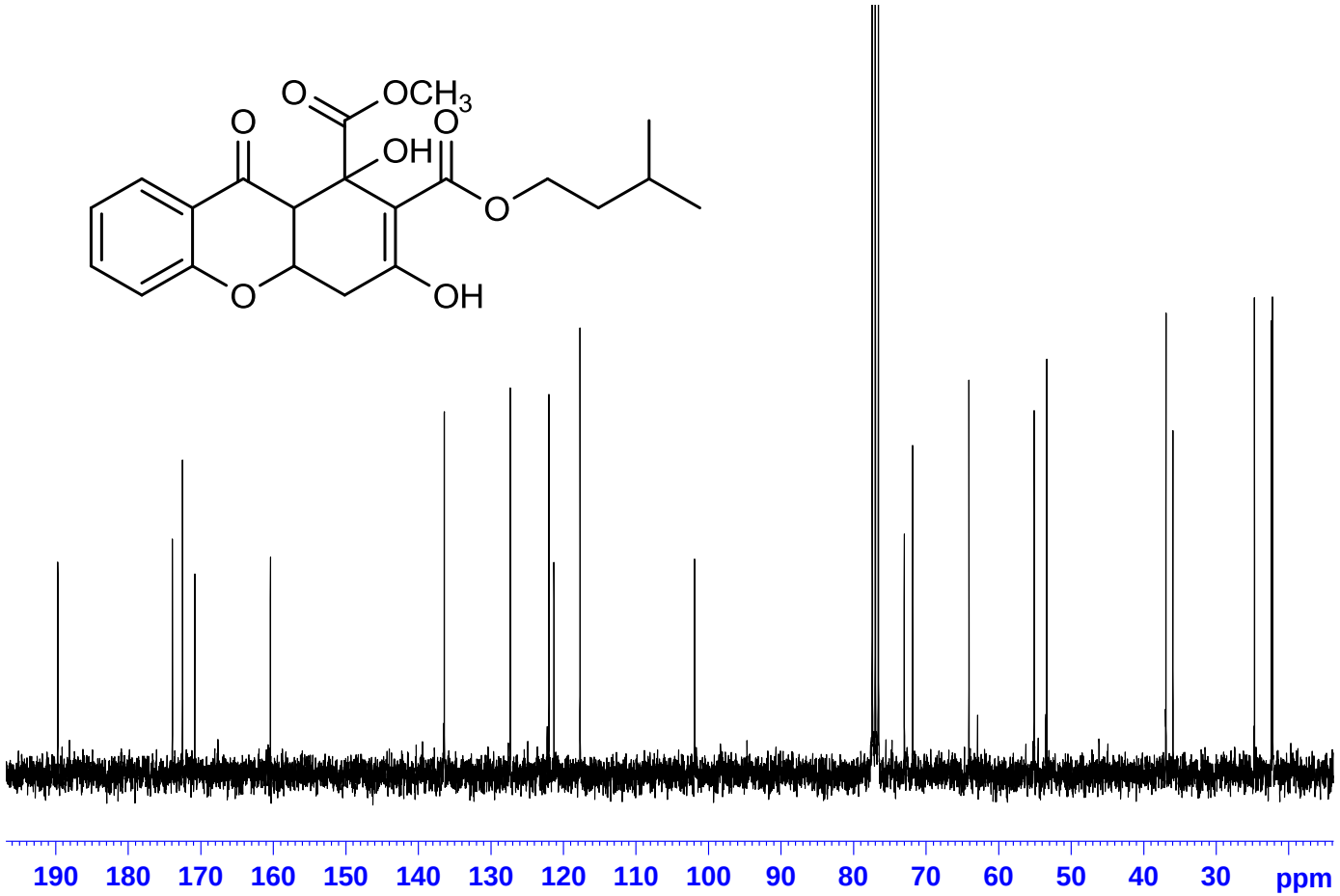
F2 - Acquisition Parameters
Date_ 20100606
Time 12.22
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 4
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 143.7
DW 81.000 usec
DE 6.00 usec
TE 295.6 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 0.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300153 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

Bunescu/ AB 290
Au13C CDCl3 /opt/topspin 1006 12

189.73 173.93 172.53 170.84 160.41 136.44 127.34 122.00 121.31 117.72 101.93 73.01 71.86 64.10 55.09 53.36 36.93 35.97 24.72 22.40 22.23



Current Data Parameters
NAME AB290 13
EXPNO 11
PROCNO 1

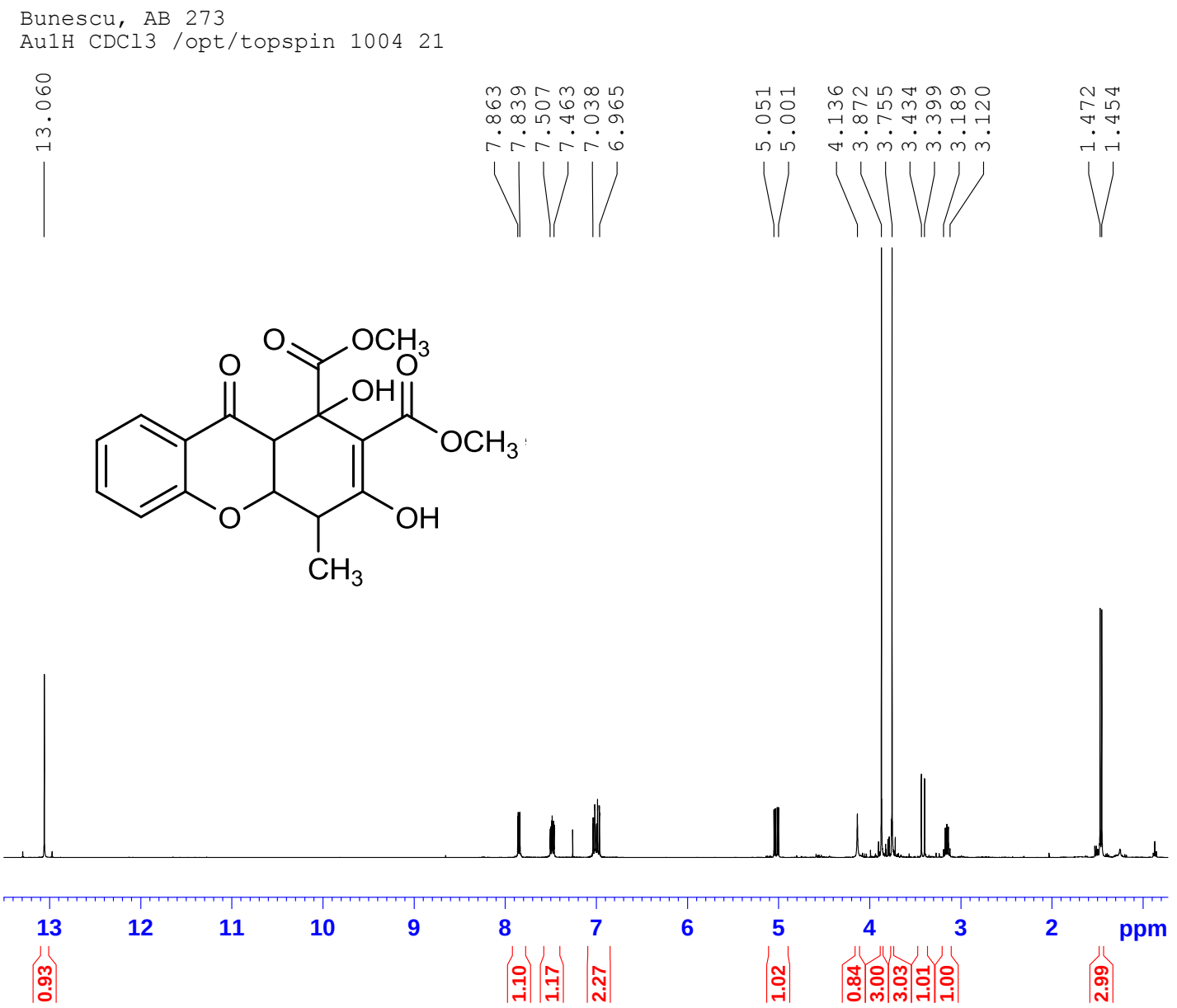
F2 - Acquisition Parameters
Date_ 20100606
Time 12.35
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 256
DS 4
SWH 21097.047 Hz
FIDRES 0.643831 Hz
AQ 0.7766516 sec
RG 32768
DW 23.700 usec
DE 6.00 usec
TE 296.1 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.60 usec
PL1 -1.10 dB
SFO1 75.4771825 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 19.00 dB
PL13 21.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677545 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Bunescu, AB 273
Au1H CDCl3 /opt/topspin 1004 21



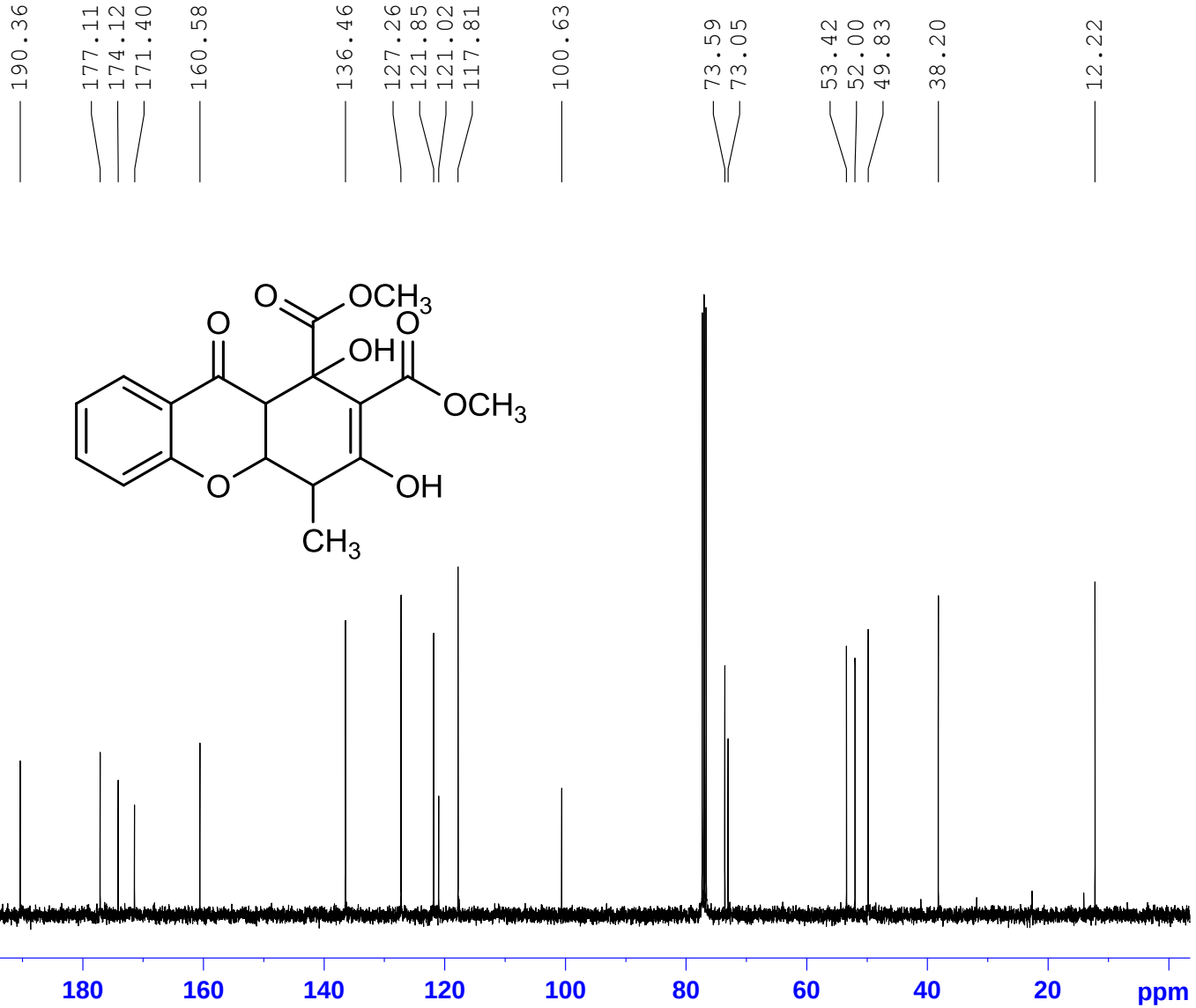
Current Data Parameters
NAME AB273 1H
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100422
Time 23.22
INSTRUM AV400
PROBHD 5 mm QNP 1H/29
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 4
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447731 sec
RG 161.3
DW 62.400 usec
DE 6.00 usec
TE 297.0 K
D1 1.50000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.50 usec
PL1 -1.00 dB
SFO1 400.1324000 MHz

F2 - Processing parameters
SI 32768
SF 400.1300210 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

Bunescu, AB 273
Au13C CDCl3 /opt/topspin 1004 21



Current Data Parameters
NAME AB273 13C
EXPNO 11
PROCNO 1

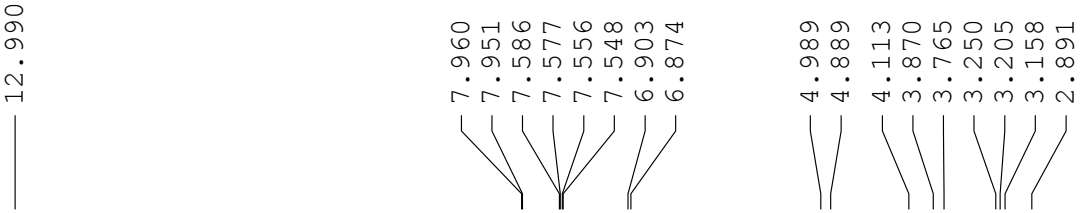
F2 - Acquisition Parameters
Date_ 20100422
Time 23.35
INSTRUM AV400
PROBHD 5 mm QNP 1H/29
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 29940.119 Hz
FIDRES 0.456850 Hz
AQ 1.0945013 sec
RG 7298.2
DW 16.700 usec
DE 6.00 usec
TE 297.0 K
D1 1.70000005 sec
d11 0.03000000 sec
DELTA 1.60000002 sec
TD0 1

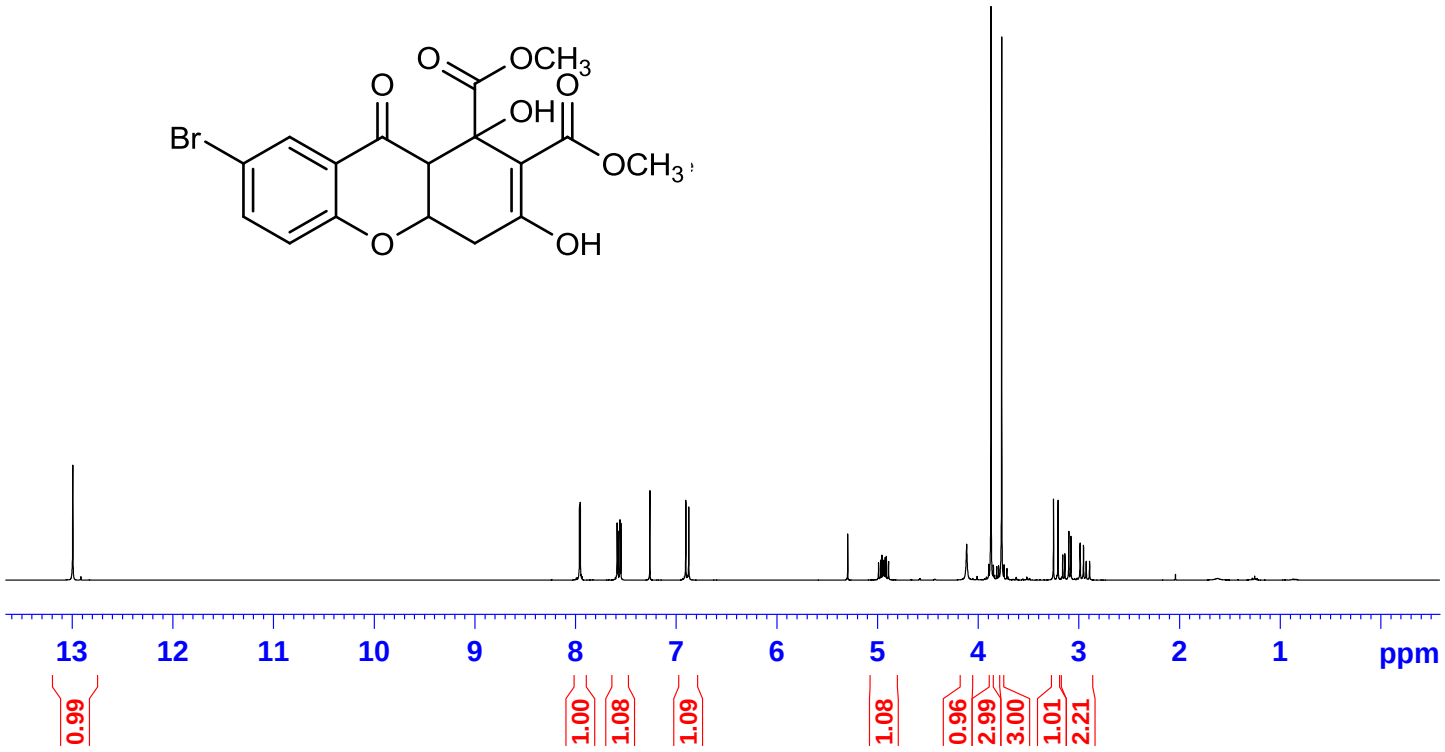
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 -1.00 dB
SFO1 100.6260690 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -1.00 dB
PL12 19.00 dB
PL13 21.00 dB
SFO2 400.1318000 MHz

F2 - Processing parameters
SI 32768
SF 100.6127758 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

Bunescu/ AB 363 A
Au1H CDCl3 /opt/topspin 1012 8





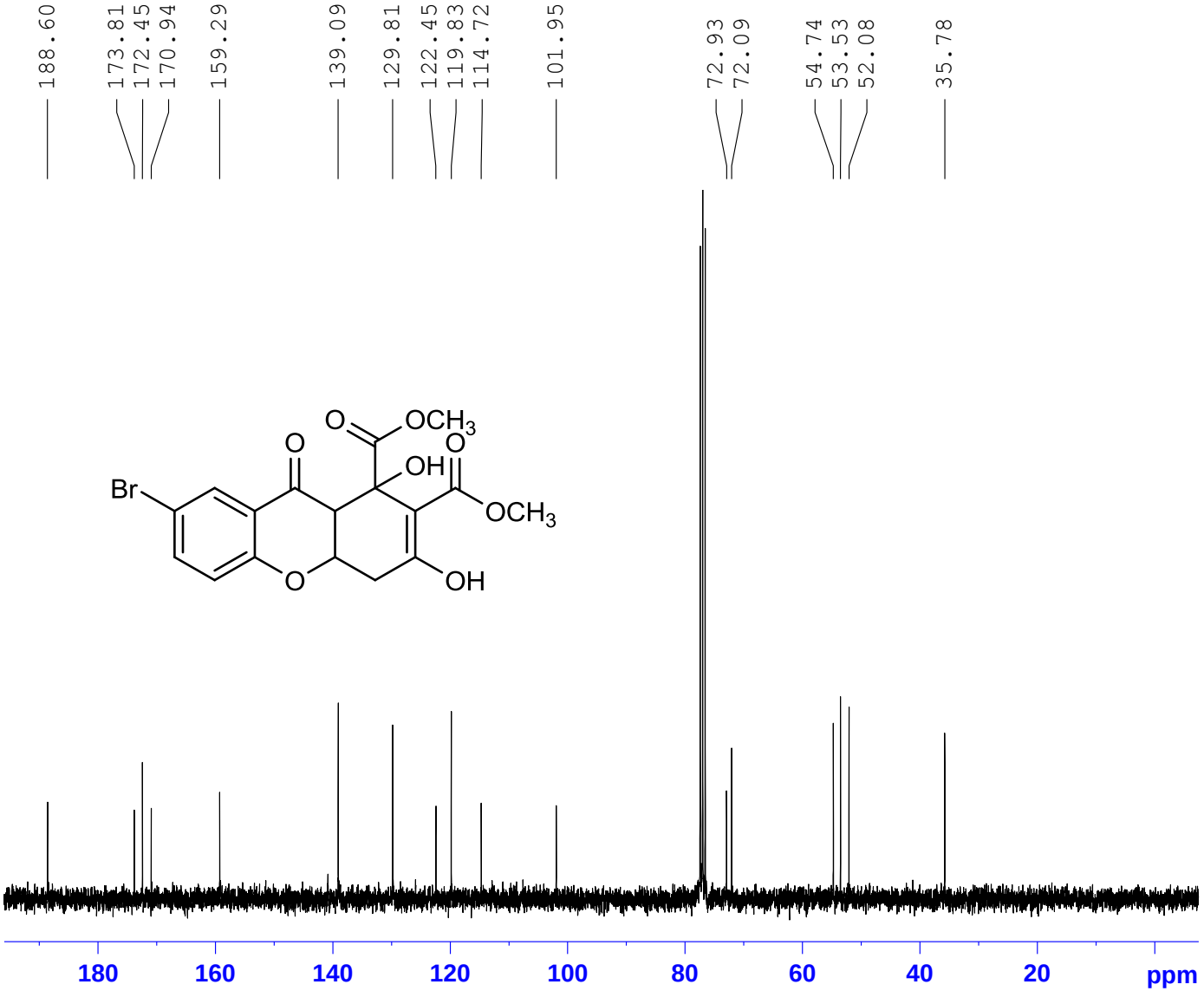
Current Data Parameters
NAME AB363A
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20101216
Time 16.18
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 4
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 322.5
DW 81.000 usec
DE 6.00 usec
TE 294.7 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 0.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300151 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

Bunescu/ AB 363 A
Au13C CDCl3 /opt/topspin 1012 8



Current Data Parameters
NAME AB363A
EXPNO 11
PROCNO 1

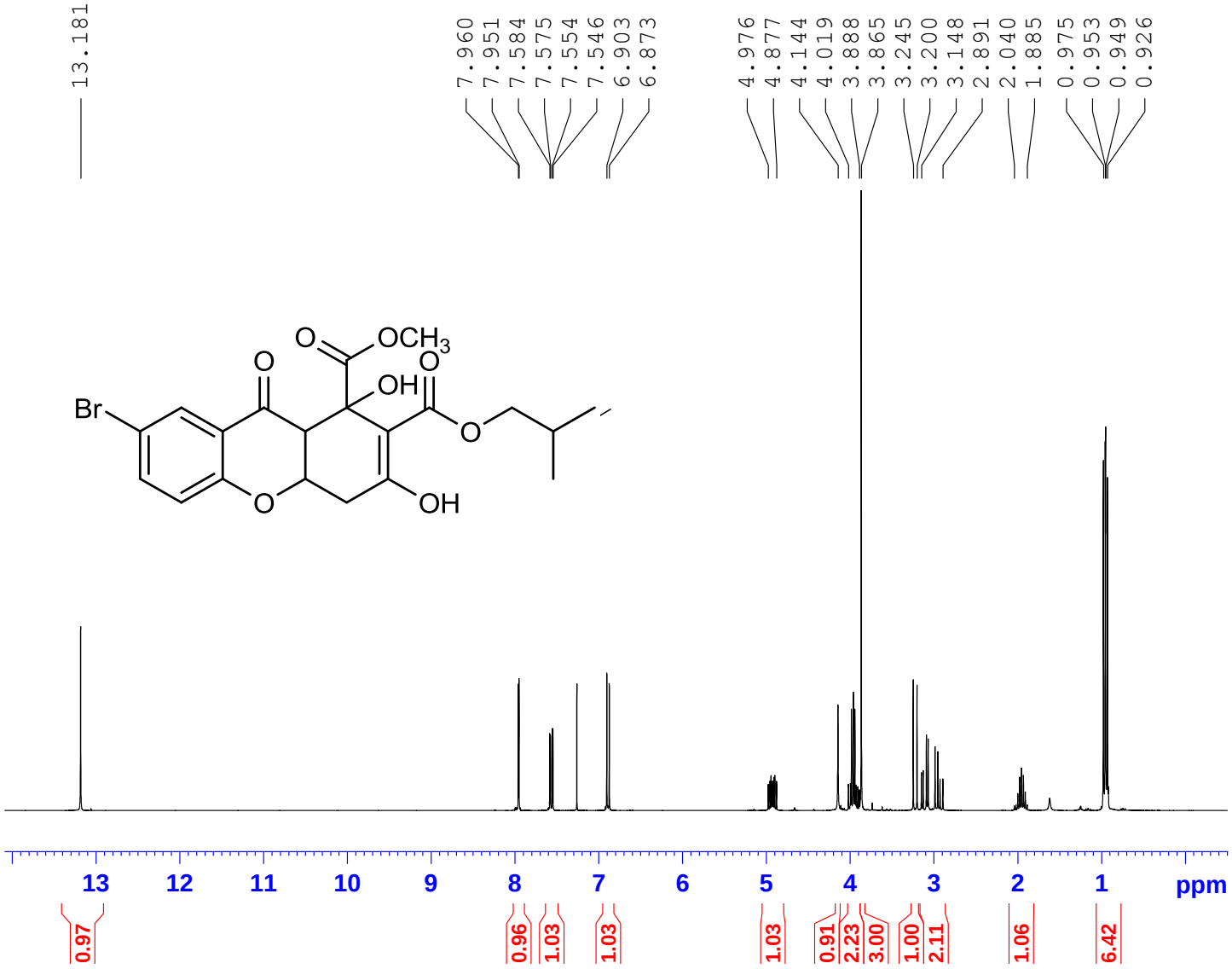
F2 - Acquisition Parameters
Date_ 20101216
Time 16.32
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 256
DS 4
SWH 21097.047 Hz
FIDRES 0.643831 Hz
AQ 0.7766516 sec
RG 32768
DW 23.700 usec
DE 6.00 usec
TE 295.3 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.60 usec
PL1 -1.10 dB
SFO1 75.4771825 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 19.00 dB
PL13 21.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677544 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

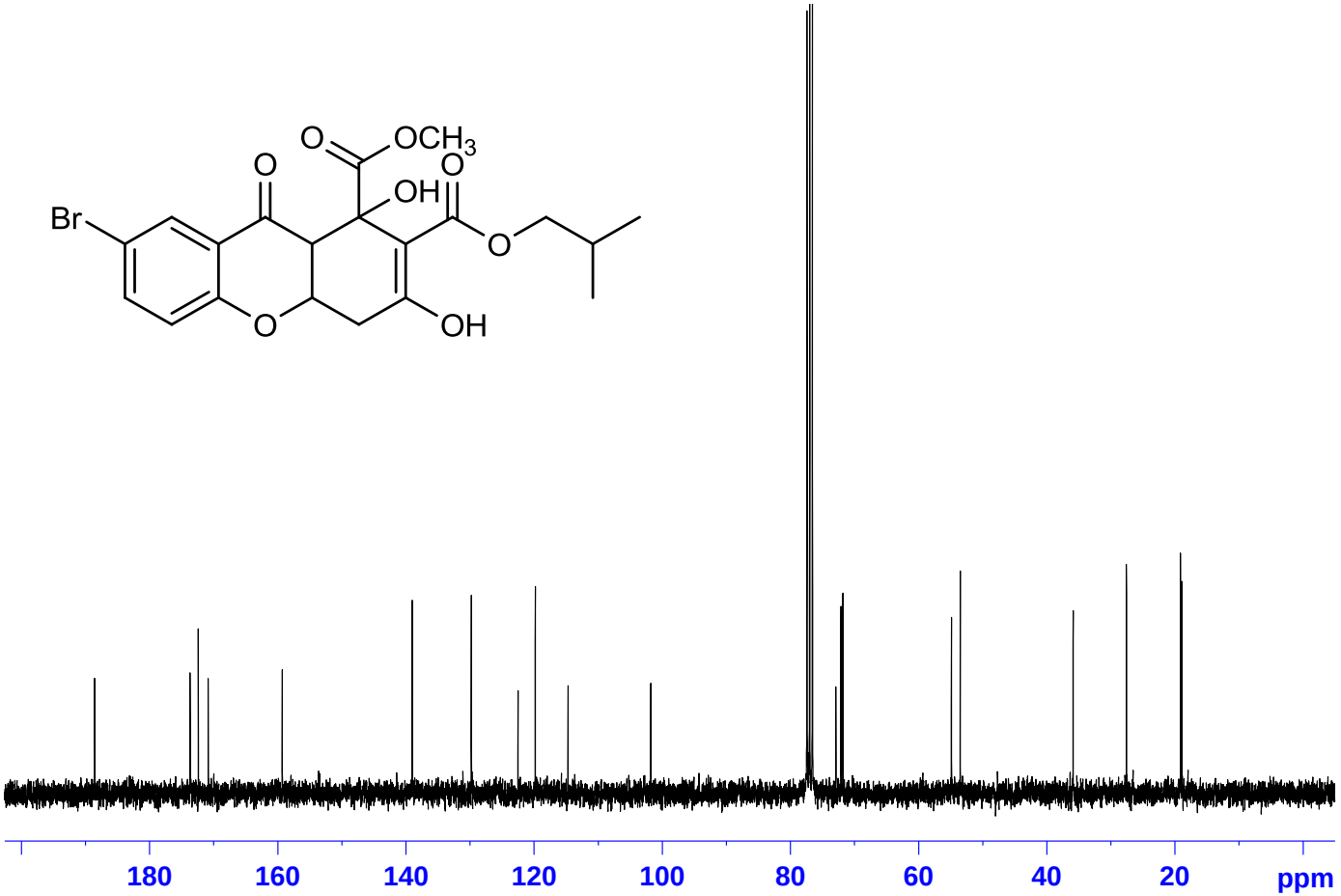
Bunescu AB373A
Au1H CDCl3 /opt/topspin 1102 50




```
Current Data Parameters
NAME                  AB373A
EXPNO                     10
PROCNO                     1

F2 - Acquisition Parameters
Date_              20110206
Time                  15.43
INSTRUM               AV300
PROBHD    5 mm PABBO BB-
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       32
DS                        4
SWH                6172.839 Hz
FIDRES             0.188380 Hz
AQ                 2.6542580 sec
RG                     161.3
DW                  81.000 usec
DE                   6.00 usec
TE                   294.3 K
D1                1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                    11.00 usec
PL1                    0.00 dB
SFO1           300.1318534 MHz

F2 - Processing parameters
SI                        32768
SF             300.1300152 MHz
WDW                        EM
SSB                         0
LB                     0.10 Hz
GB                          0
PC                      1.00
```

Bunescu AB373A
Au13C CDCl3 /opt/topspin 1102 50

188.56 173.67 172.39 170.86 159.28 139.04 129.81 122.53 119.83 114.71 101.81 72.92 72.14 71.83 54.89 53.51 35.89 27.57 19.13 18.92



Current Data Parameters
NAME AB373A
EXPNO 11
PROCNO 1

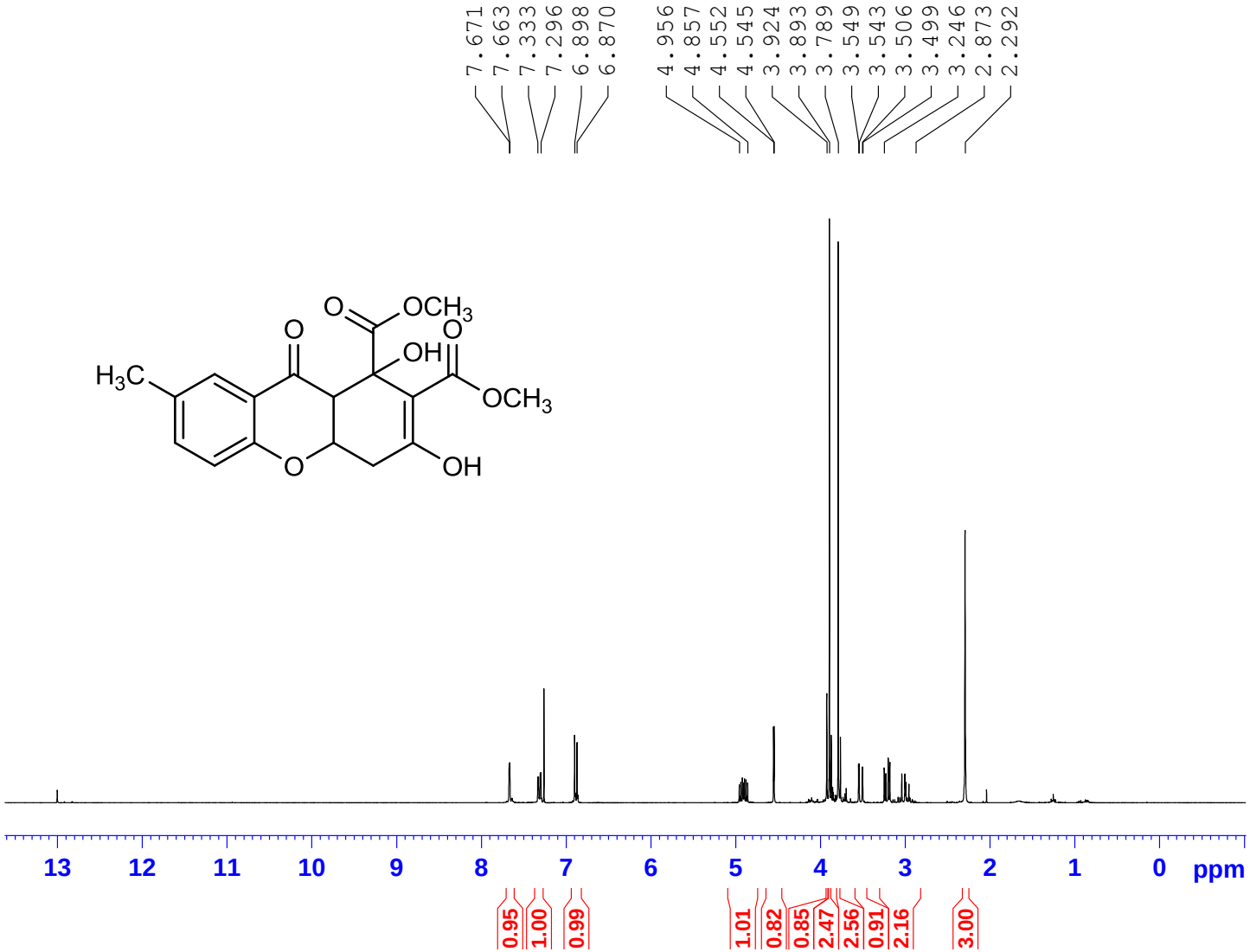
F2 - Acquisition Parameters
Date_ 20110206
Time 15.58
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 256
DS 4
SWH 21097.047 Hz
FIDRES 0.643831 Hz
AQ 0.7766516 sec
RG 32768
DW 23.700 usec
DE 6.00 usec
TE 294.8 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.60 usec
PL1 -1.10 dB
SFO1 75.4771825 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 19.00 dB
PL13 21.00 dB
SFO2 300.1312005 MHz

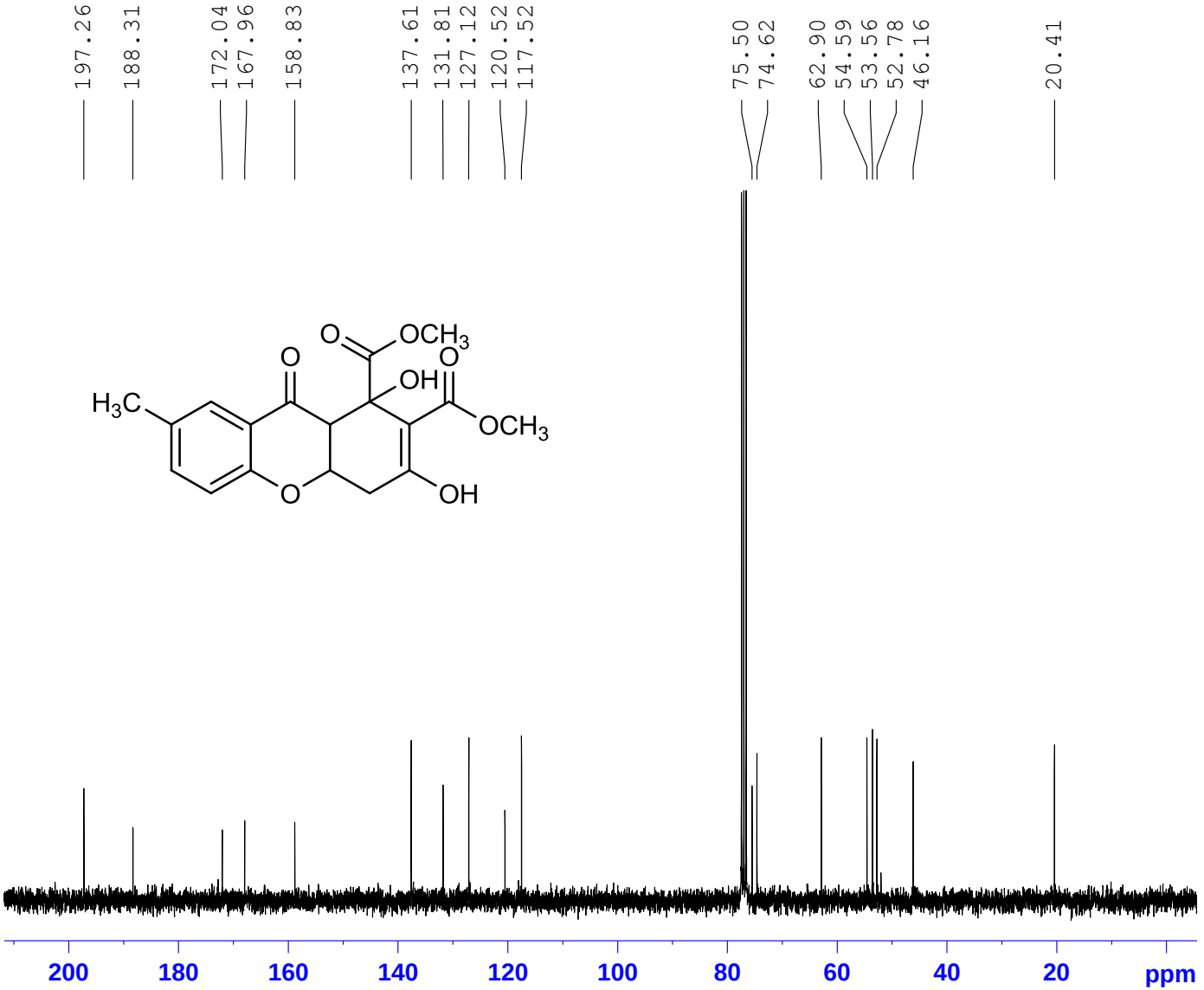
F2 - Processing parameters
SI 32768
SF 75.4677539 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Bunescu AB367A
Au1H CDCl3 /opt/topspin 1105 28




```
Current Data Parameters
NAME          AC367A 1H
EXPNO                10
PROCNO                1

F2 - Acquisition Parameters
Date_          20110505
Time              23.36
INSTRUM           AV300
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   32
DS                    4
SWH            6172.839 Hz
FIDRES         0.188380 Hz
AQ             2.6542580 sec
RG                322.5
DW               81.000 usec
DE                6.00 usec
TE               295.6 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                11.00 usec
PL1                0.00 dB
SFO1        300.1318534 MHz

F2 - Processing parameters
SI                32768
SF          300.1300152 MHz
WDW                  EM
SSB                   0
LB                 0.10 Hz
GB                    0
PC                 1.00
```

Bunescu AB367A
Au13C CDCl3 /opt/topspin 1105 28



Current Data Parameters
NAME AB367A 13C
EXPNO 11
PROCNO 1

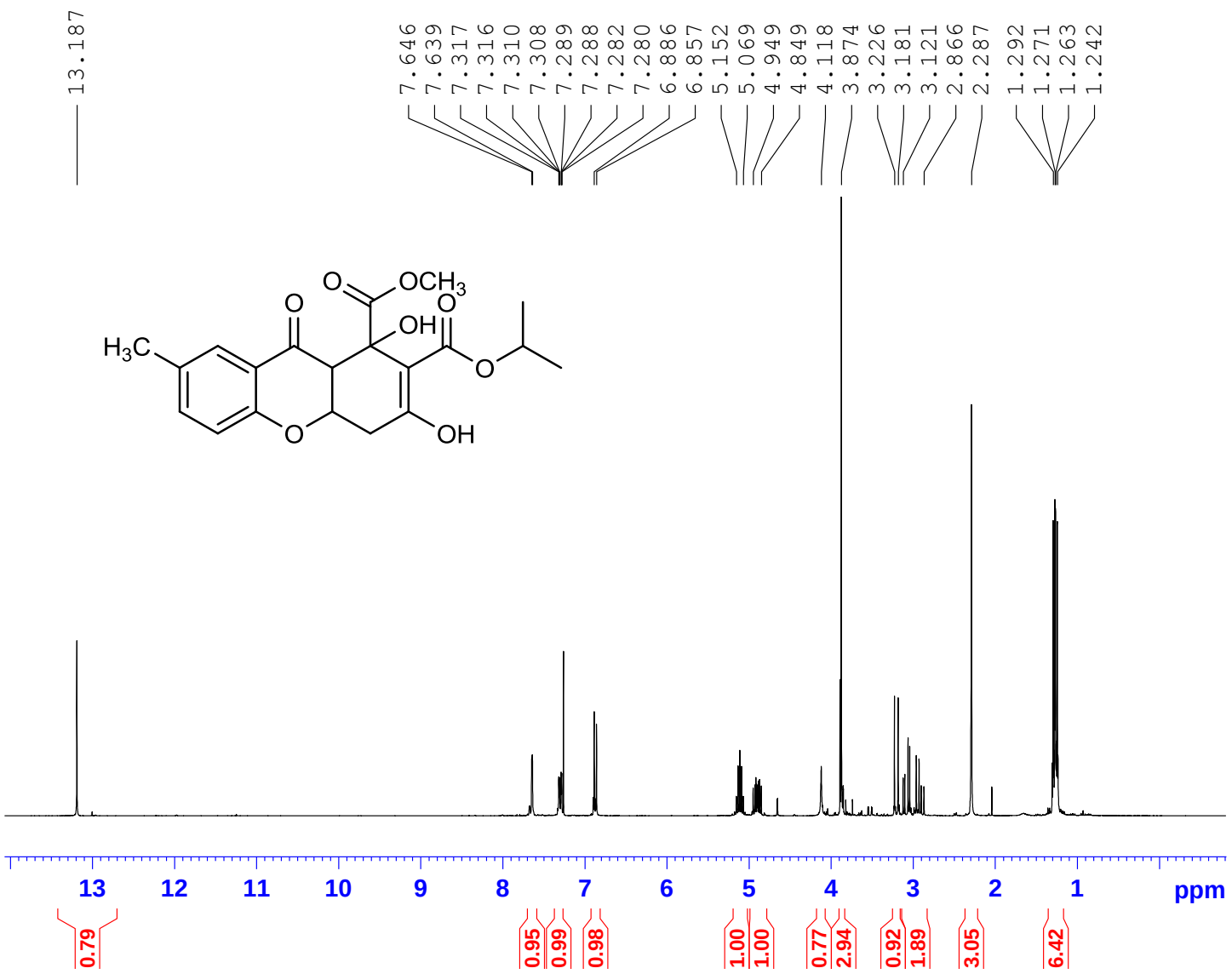
F2 - Acquisition Parameters
Date_ 20110505
Time 23.49
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 256
DS 4
SWH 21097.047 Hz
FIDRES 0.643831 Hz
AQ 0.7766516 sec
RG 32768
DW 23.700 usec
DE 6.00 usec
TE 296.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.60 usec
PL1 -1.10 dB
SFO1 75.4771825 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 19.00 dB
PL13 21.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677540 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Bunescu/ AB 385
Au1H CDCl3 /opt/topspin 1102 26



Current Data Parameters
NAME AB385A
EXPNO 10
PROCNO 1

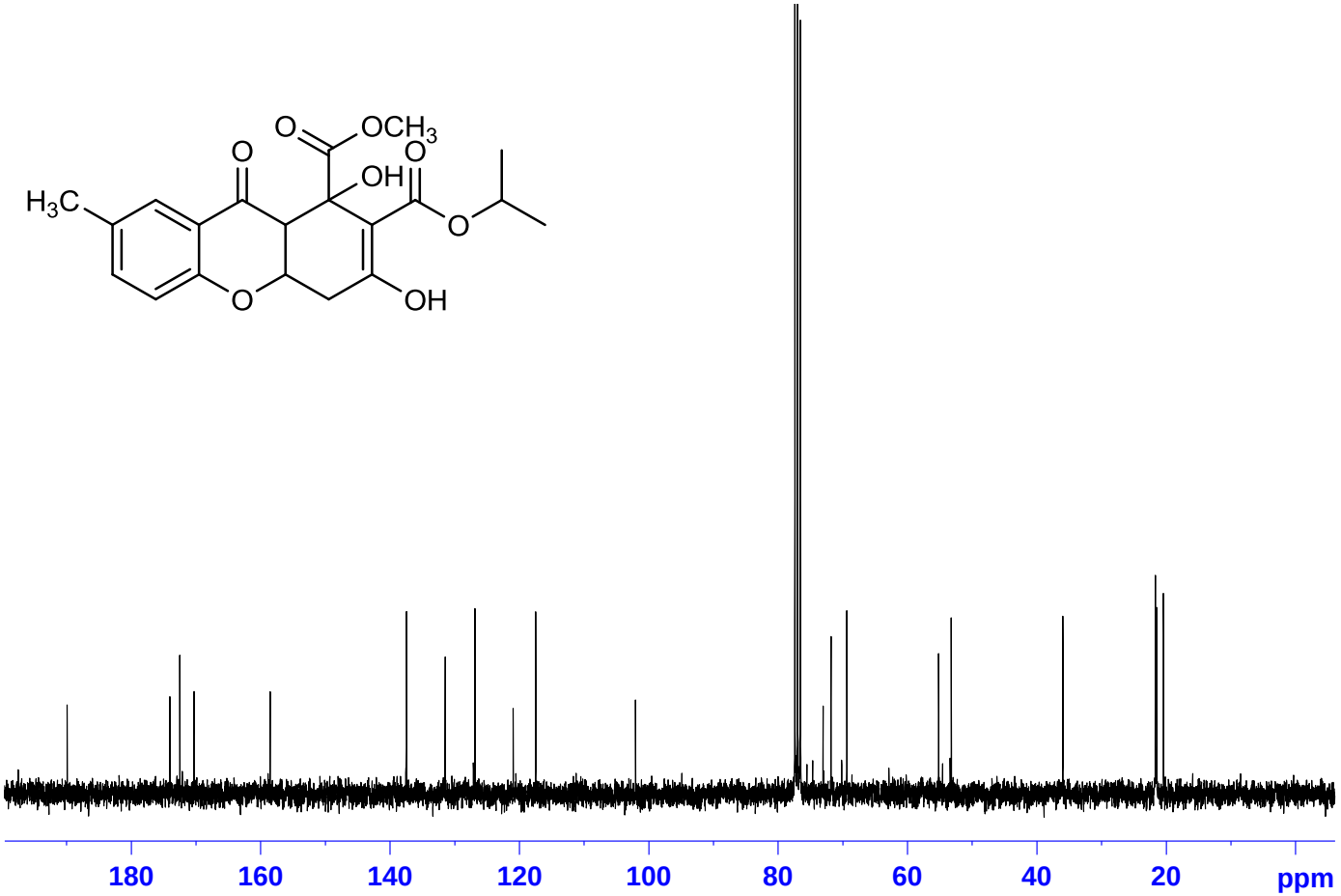
F2 - Acquisition Parameters
Date_ 20110218
Time 20.08
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 4
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 181
DW 81.000 usec
DE 6.00 usec
TE 294.4 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 0.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300152 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

Bunescu/ AB 385
Au13C CDCl3 /opt/topspin 1102 26

189.90 174.00 172.47 170.29 158.51 137.47 131.49 126.88 120.97 117.49 102.07 71.83 69.39 55.19 53.23 35.98 21.68 21.45 20.42



Current Data Parameters
NAME AB385A
EXPNO 11
PROCNO 1

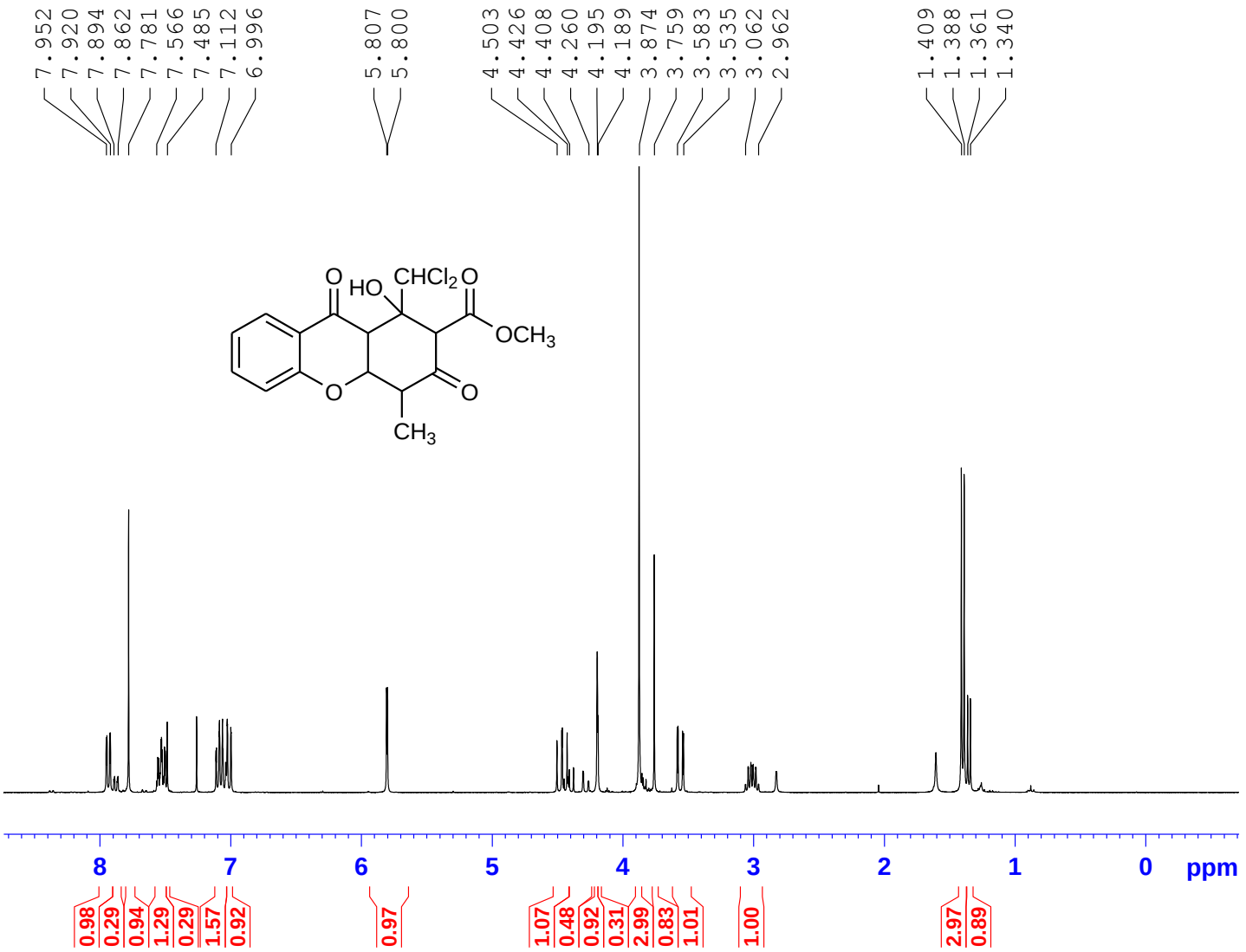
F2 - Acquisition Parameters
Date_ 20110218
Time 20.21
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 256
DS 4
SWH 21097.047 Hz
FIDRES 0.643831 Hz
AQ 0.7766516 sec
RG 32768
DW 23.700 usec
DE 6.00 usec
TE 294.8 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.60 usec
PL1 -1.10 dB
SFO1 75.4771825 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 19.00 dB
PL13 21.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677542 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

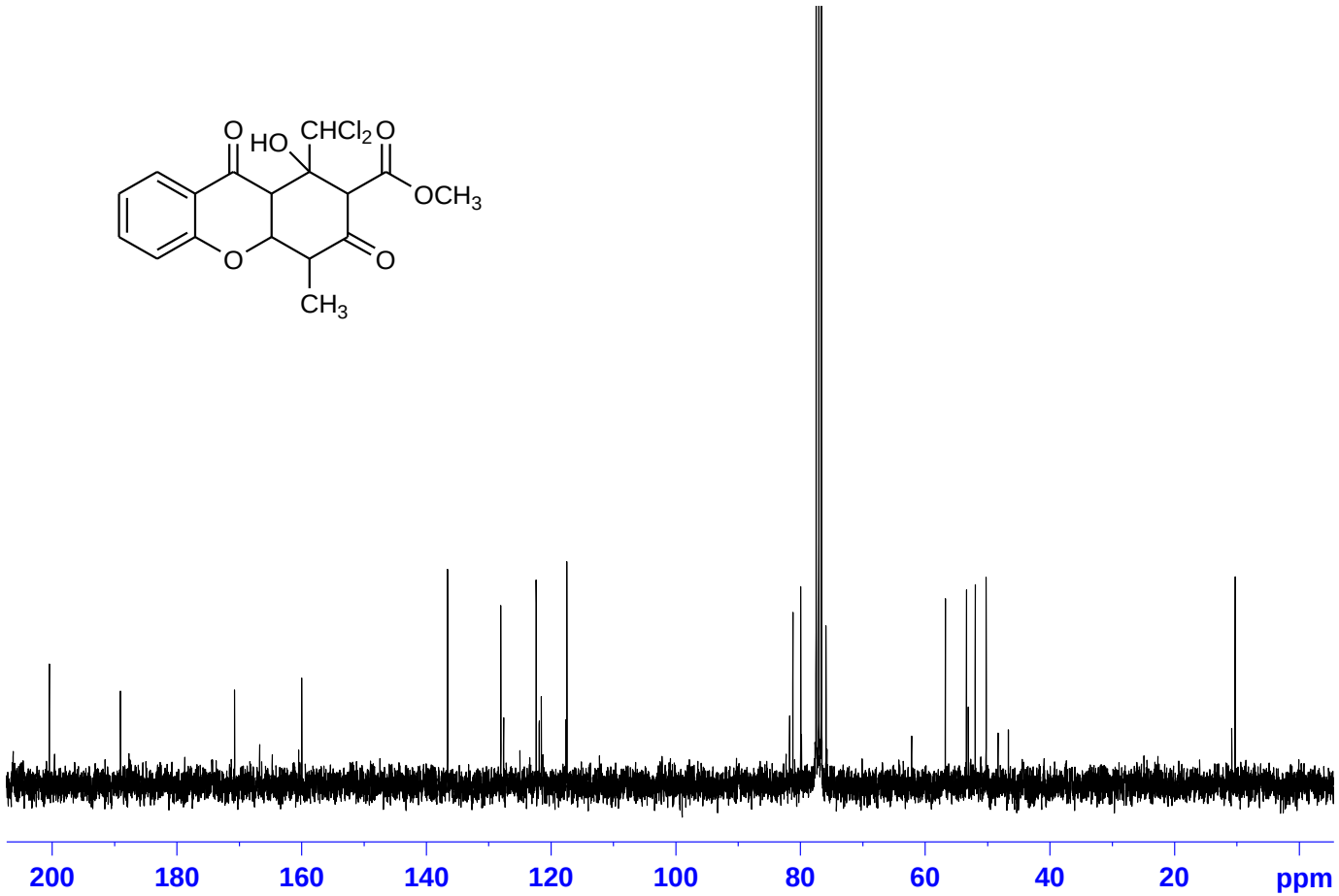
Bunescu, AB 275
Au1H CDCl3 /opt/topspin 1004 28




```
Current Data Parameters
NAME                  AB275 1H
EXPNO                       10
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20100501
Time                      3.10
INSTRUM                 AV300
PROBHD    5 mm PABBO BB-
PULPROG                   zg30
TD                       32768
SOLVENT                  CDCl3
NS                          32
DS                           4
SWH                  6172.839 Hz
FIDRES               0.188380 Hz
AQ                   2.6542580 sec
RG                       322.5
DW                      81.000 usec
DE                       6.00 usec
TE                       294.8 K
D1                1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        1H
P1                      11.00 usec
PL1                      0.00 dB
SFO1             300.1318534 MHz

F2 - Processing parameters
SI                       32768
SF                300.1300153 MHz
WDW                         EM
SSB                          0
LB                       0.10 Hz
GB                           0
PC                       1.00
```

Bunescu, AB 275
Au13C CDCl3 /opt/topspin 1004 28

200.49 189.08 170.79 160.01 136.62 128.08 127.63 122.40 121.91 121.57 117.60 117.48 81.76 81.23 79.97 79.84 75.92 62.18 56.76 53.38 53.10 51.98 50.23 48.34 46.67 10.84 10.29



Current Data Parameters
NAME AB275 13C
EXPNO 11
PROCNO 1

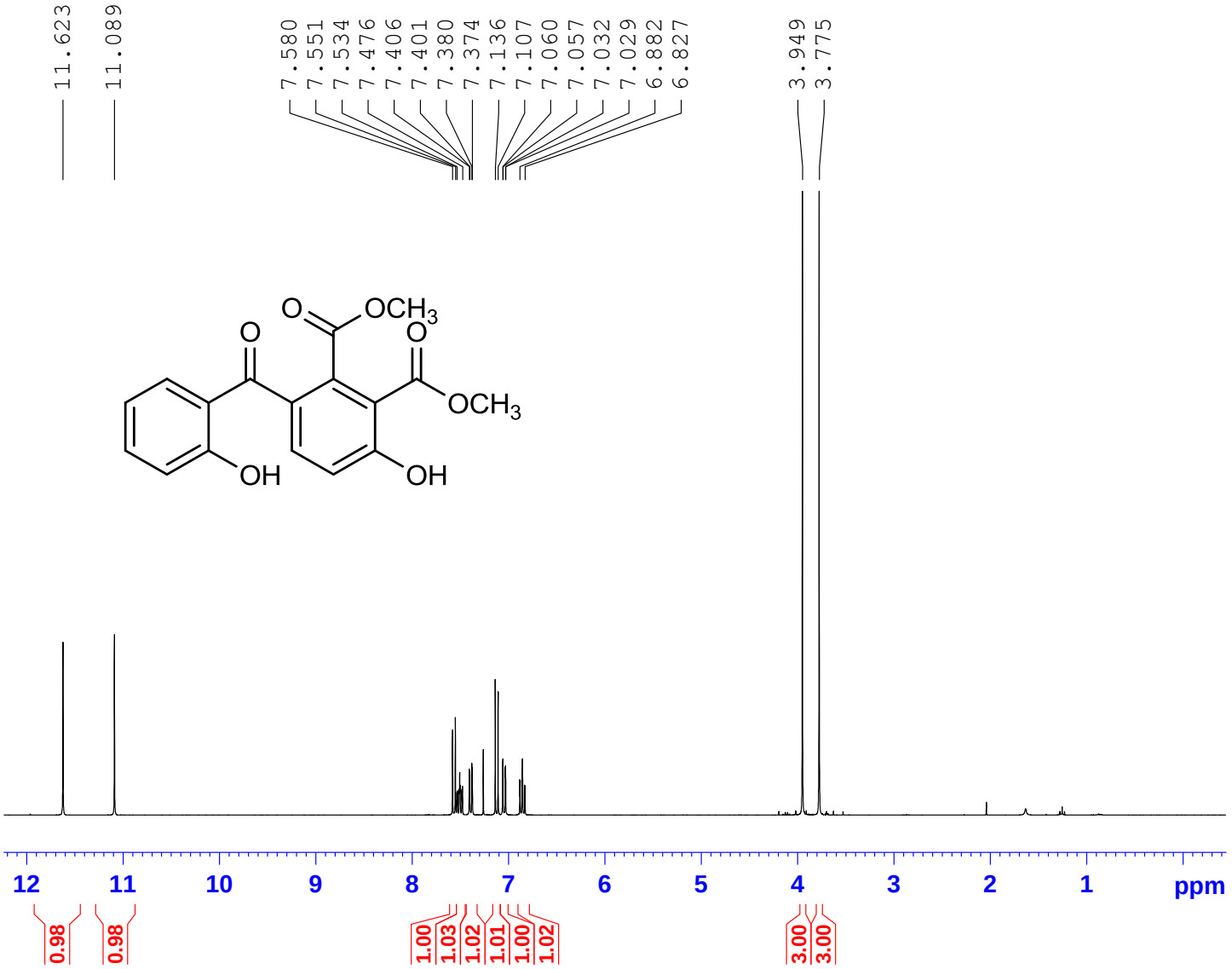
F2 - Acquisition Parameters
Date_ 20100501
Time 3.25
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 256
DS 4
SWH 21097.047 Hz
FIDRES 0.643831 Hz
AQ 0.7766516 sec
RG 32768
DW 23.700 usec
DE 6.00 usec
TE 295.4 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.60 usec
PL1 -1.10 dB
SFO1 75.4771825 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 19.00 dB
PL13 21.00 dB
SFO2 300.1312005 MHz

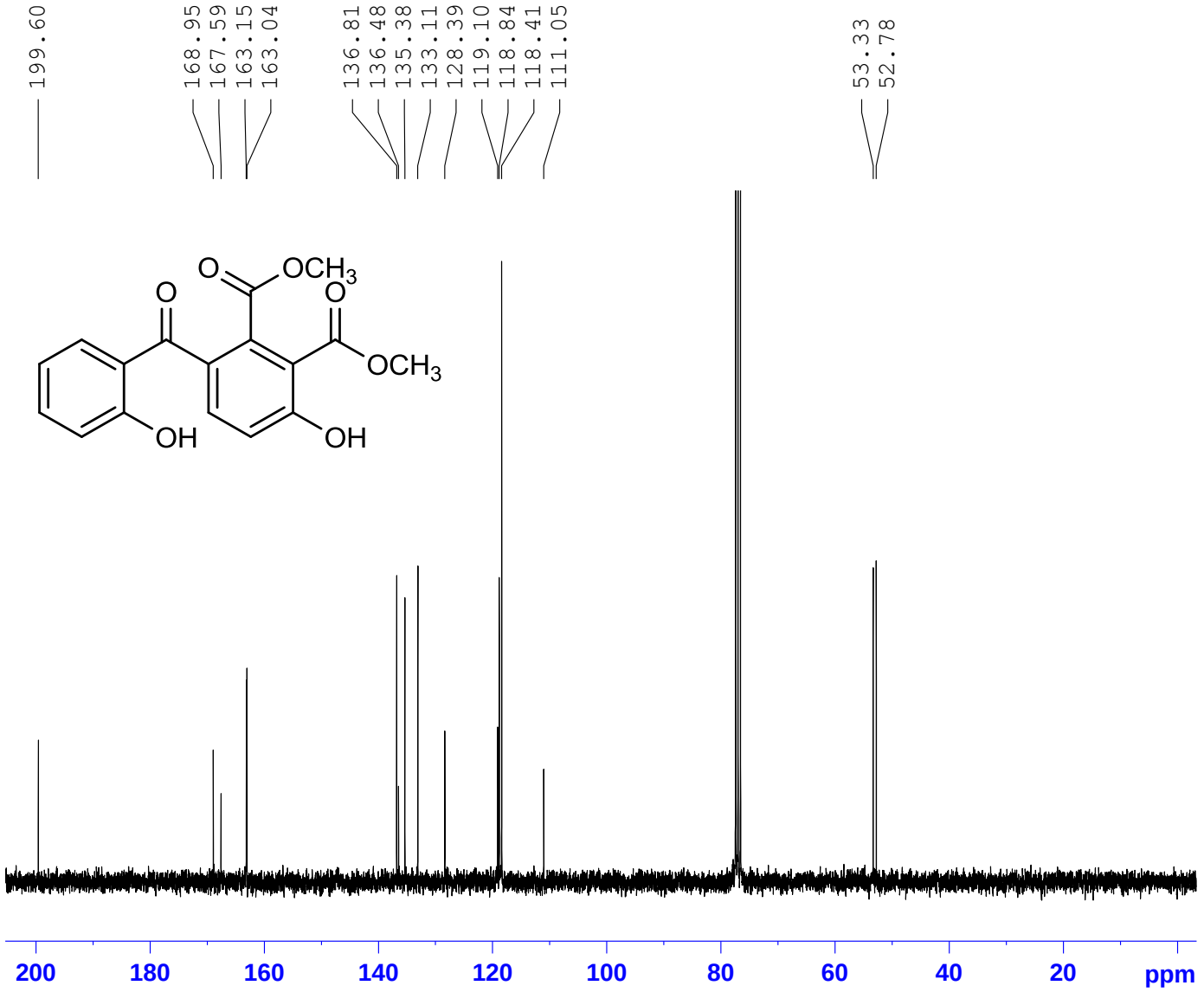
F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Bunescu/ AB 310
Au1H CDCl3 /opt/topspin 1008 12




```
Current Data Parameters
NAME            AB310 1H
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20100831
Time               11.05
INSTRUM            AV300
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     4
SWH             6172.839 Hz
FIDRES          0.188380 Hz
AQ              2.6542580 sec
RG                   181
DW                81.000 usec
DE                 6.00 usec
TE                295.8 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.00 usec
PL1                 0.00 dB
SFO1         300.1318534 MHz

F2 - Processing parameters
SI                 32768
SF           300.1300152 MHz
WDW                   EM
SSB                    0
LB                  0.10 Hz
GB                     0
PC                  1.00
```

Bunescu/ AB 310
Au13C CDCl3 /opt/topspin 1008 12



Current Data Parameters
NAME AB310 13C
EXPNO 11
PROCNO 1

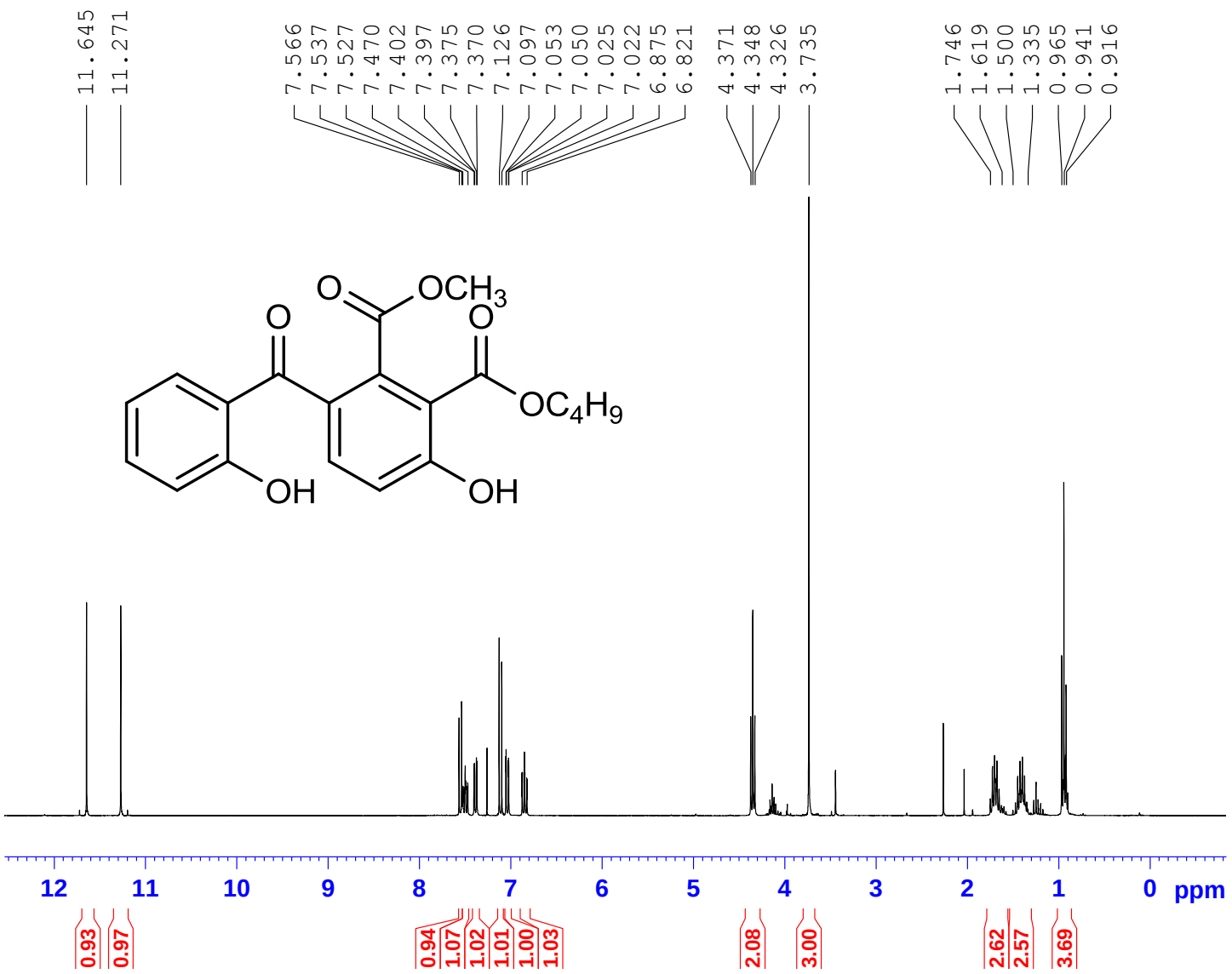
F2 - Acquisition Parameters
Date_ 20100831
Time 15.50
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 256
DS 4
SWH 21097.047 Hz
FIDRES 0.643831 Hz
AQ 0.7766516 sec
RG 32768
DW 23.700 usec
DE 6.00 usec
TE 296.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.60 usec
PL1 -1.10 dB
SFO1 75.4771825 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 19.00 dB
PL13 21.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677548 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Bumenu AB 331
Au1H CDCl3 /opt/topspin 1010 30



Current Data Parameters
NAME AB331
EXPNO 10
PROCNO 1

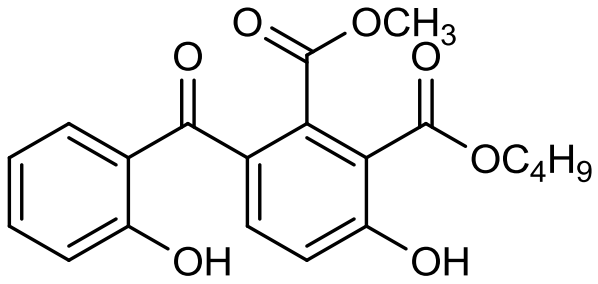
F2 - Acquisition Parameters
Date_ 20101009
Time 8.20
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 4
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 114
DW 81.000 usec
DE 6.00 usec
TE 294.6 K
D1 1.00000000 sec
TD0 1

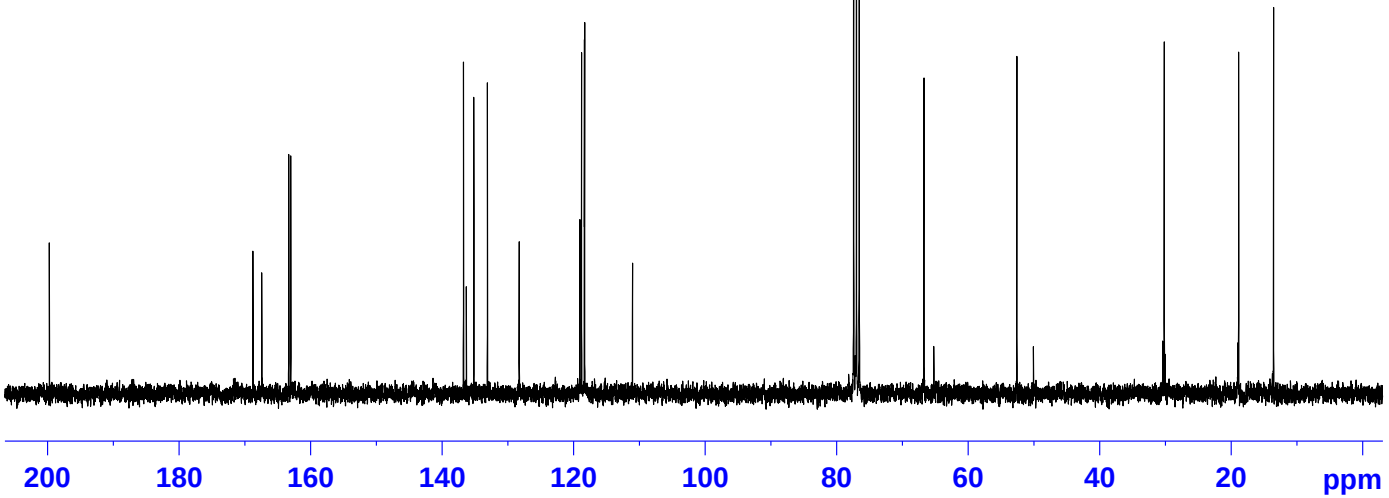
======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 0.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300153 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

Bumenu AB 331
Au13C CDCl3 /opt/topspin 1010 30

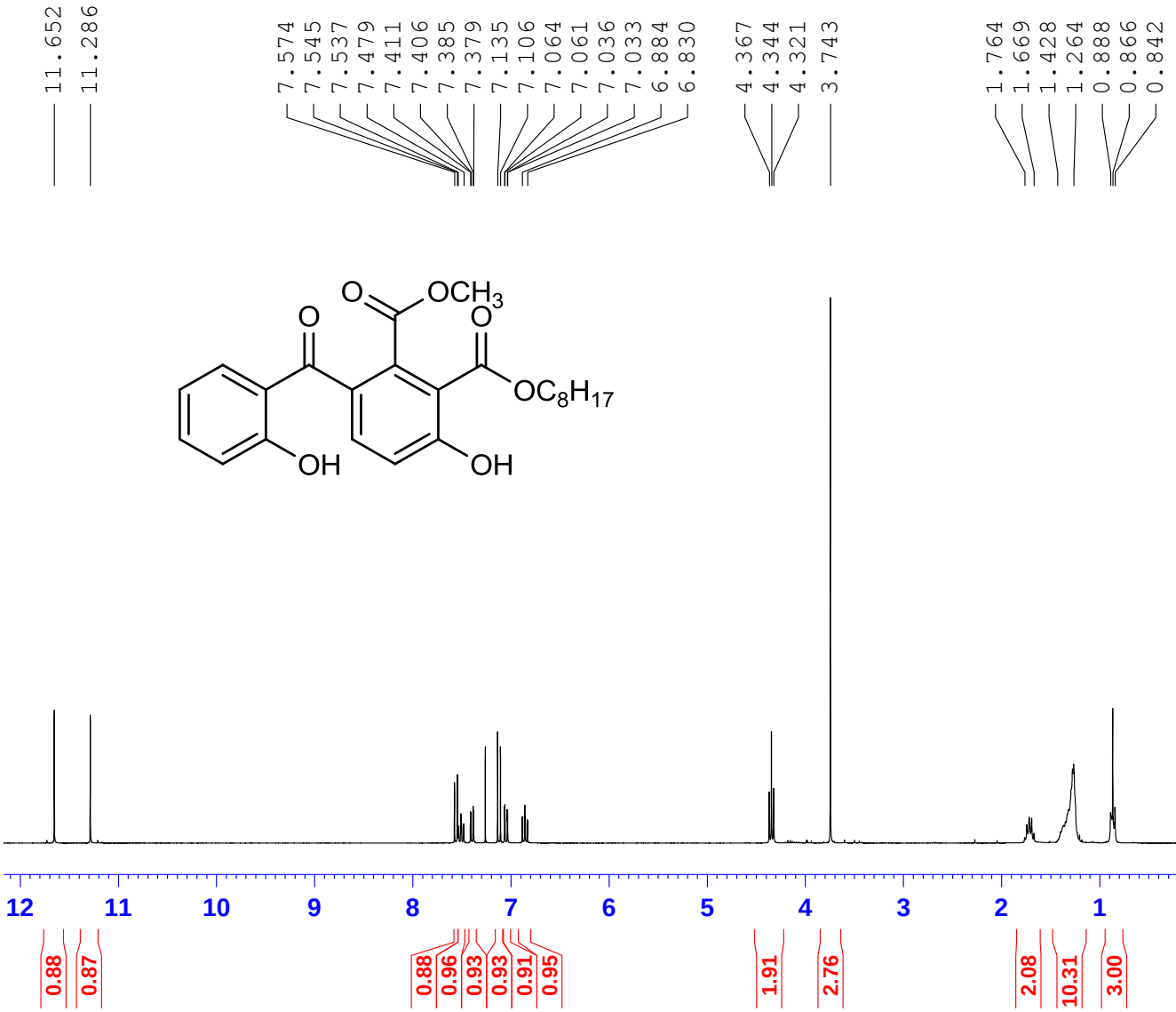
199.70
168.75
167.40
163.31
163.00
136.77
136.32
135.19
133.13
128.29
119.08
118.81
118.38
118.35
111.07
66.74
52.62
30.19
18.89
13.57






```
Current Data Parameters
NAME                 AB331 13C
EXPNO                       11
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20101009
Time                      8.33
INSTRUM                 AV300
PROBHD    5 mm PABBO BB-
PULPROG                 zgpg30
TD                       32768
SOLVENT                  CDCl3
NS                         256
DS                           4
SWH                 21097.047 Hz
FIDRES               0.643831 Hz
AQ                   0.7766516 sec
RG                       32768
DW                      23.700 usec
DE                       6.00 usec
TE                      295.1 K
D1                  2.00000000 sec
d11                 0.03000000 sec
DELTA               1.89999998 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        13C
P1                        9.60 usec
PL1                      -1.10 dB
SFO1                75.4771825 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                         1H
PCPD2                   100.00 usec
PL2                       0.00 dB
PL12                     19.00 dB
PL13                     21.00 dB
SFO2               300.1312005 MHz

F2 - Processing parameters
SI                       32768
SF                  75.4677558 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40
```

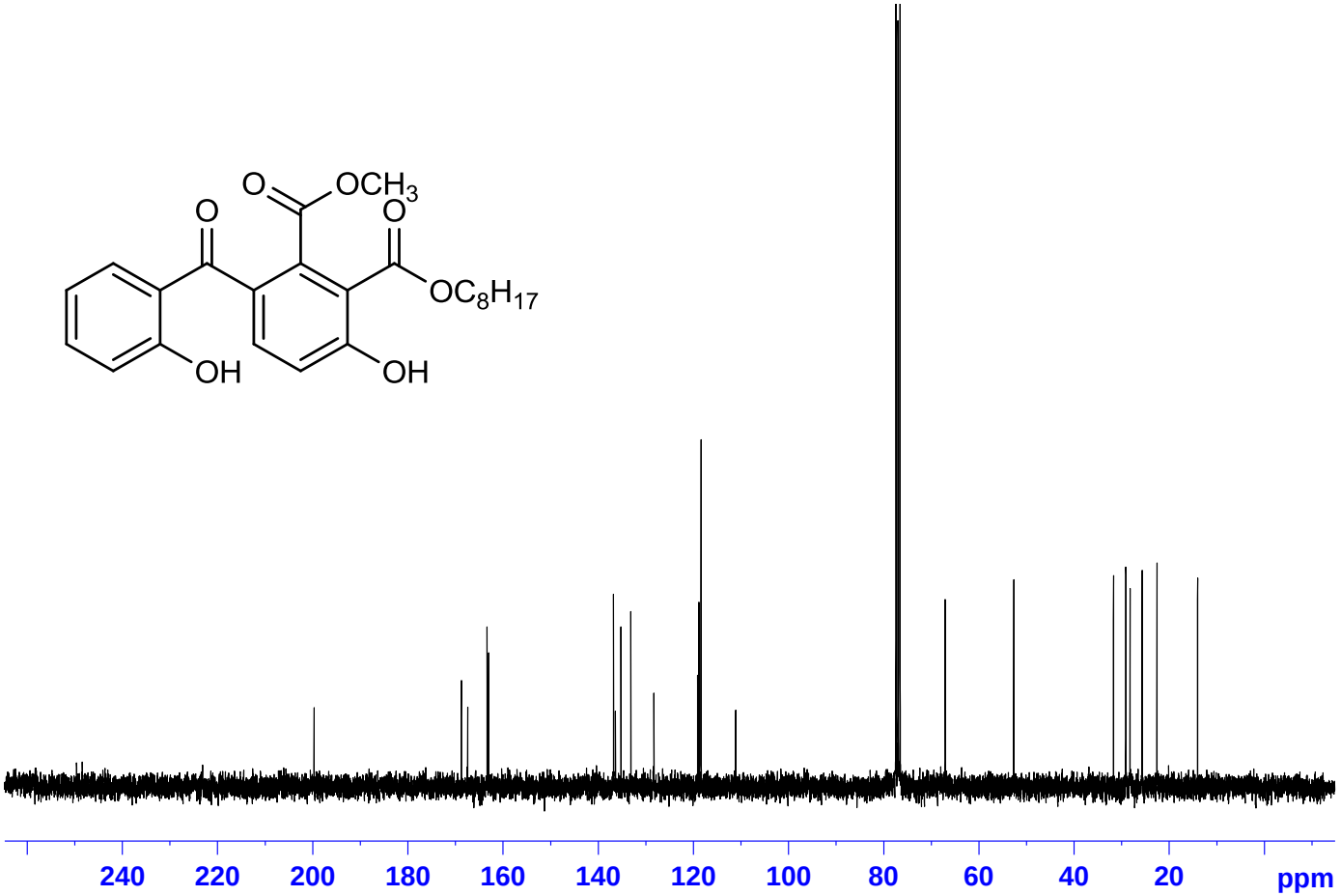
Bunescu AB333
Au1H CDCl3 /opt/topspin 1103 41




```
Current Data Parameters
NAME                  AB333
EXPNO                    10
PROCNO                    1

F2 - Acquisition Parameters
Date_              20110310
Time                   6.12
INSTRUM               AV300
PROBHD    5 mm PABBO BB-
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       32
DS                        4
SWH                6172.839 Hz
FIDRES            0.188380 Hz
AQ                2.6542580 sec
RG                    161.3
DW                  81.000 usec
DE                   6.00 usec
TE                   294.3 K
D1              1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     1H
P1                   11.00 usec
PL1                   0.00 dB
SFO1          300.1318534 MHz

F2 - Processing parameters
SI                    32768
SF            300.1300152 MHz
WDW                      EM
SSB                       0
LB                   0.10 Hz
GB                        0
PC                    1.00
```

Bunescu AB333
Au13C CDCl3 /opt/topspin 1103 41

199.72
168.78
167.44
163.36
163.05
136.80
136.40
135.25
133.16
128.31
119.11
118.84
118.40
111.12
67.10
52.66
31.72
29.16
29.10
28.25
25.70
22.58
14.04



Current Data Parameters
NAME AB333
EXPNO 11
PROCNO 1

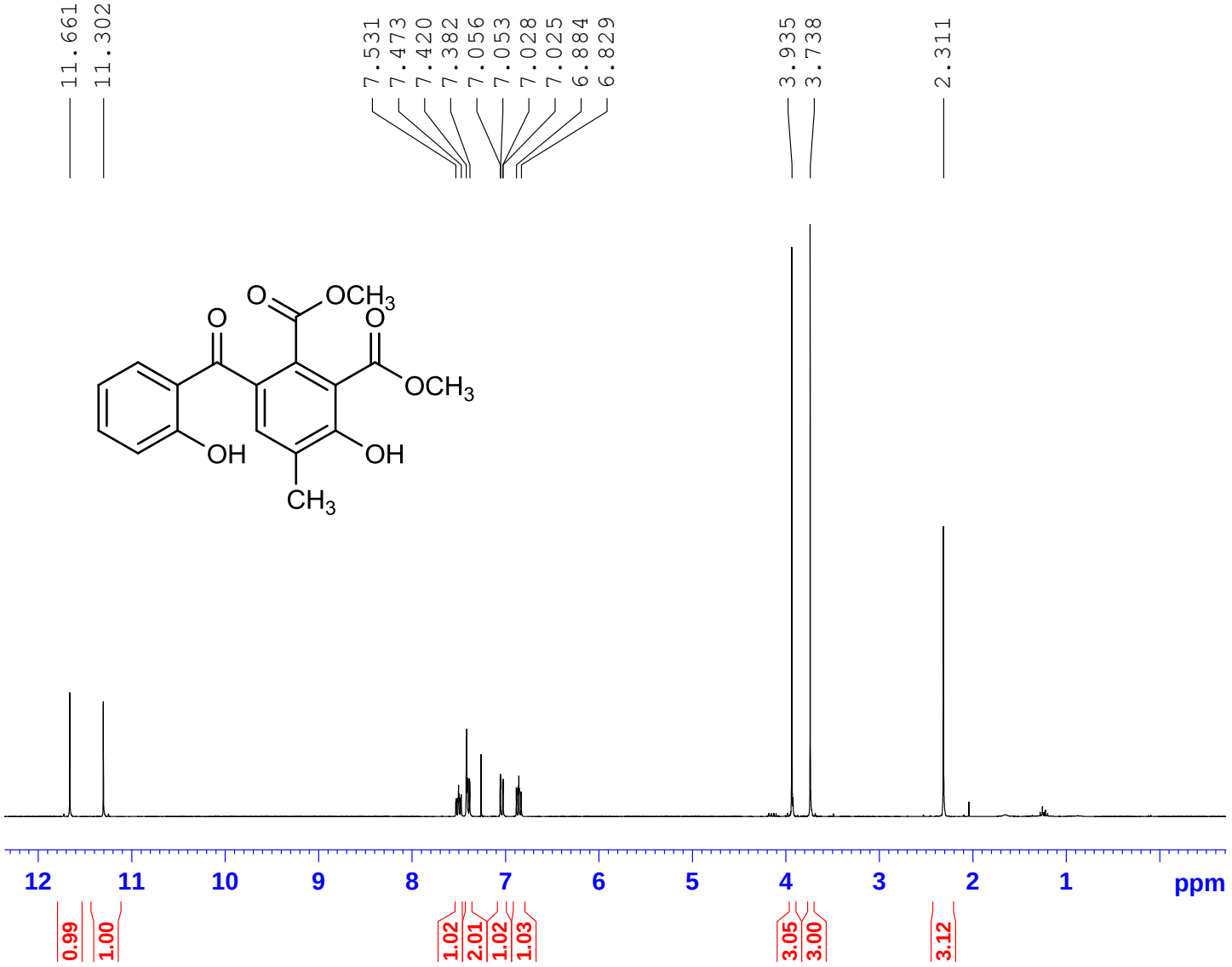
F2 - Acquisition Parameters
Date_ 20110310
Time 6.25
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 256
DS 4
SWH 21097.047 Hz
FIDRES 0.643831 Hz
AQ 0.7766516 sec
RG 32768
DW 23.700 usec
DE 6.00 usec
TE 294.8 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.60 usec
PL1 -1.10 dB
SFO1 75.4771825 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 19.00 dB
PL13 21.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677537 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Bunescu/ AB 327
Au1H CDCl3 /opt/topspin 1009 53



Current Data Parameters
NAME AB327 1H
EXPNO 10
PROCNO 1

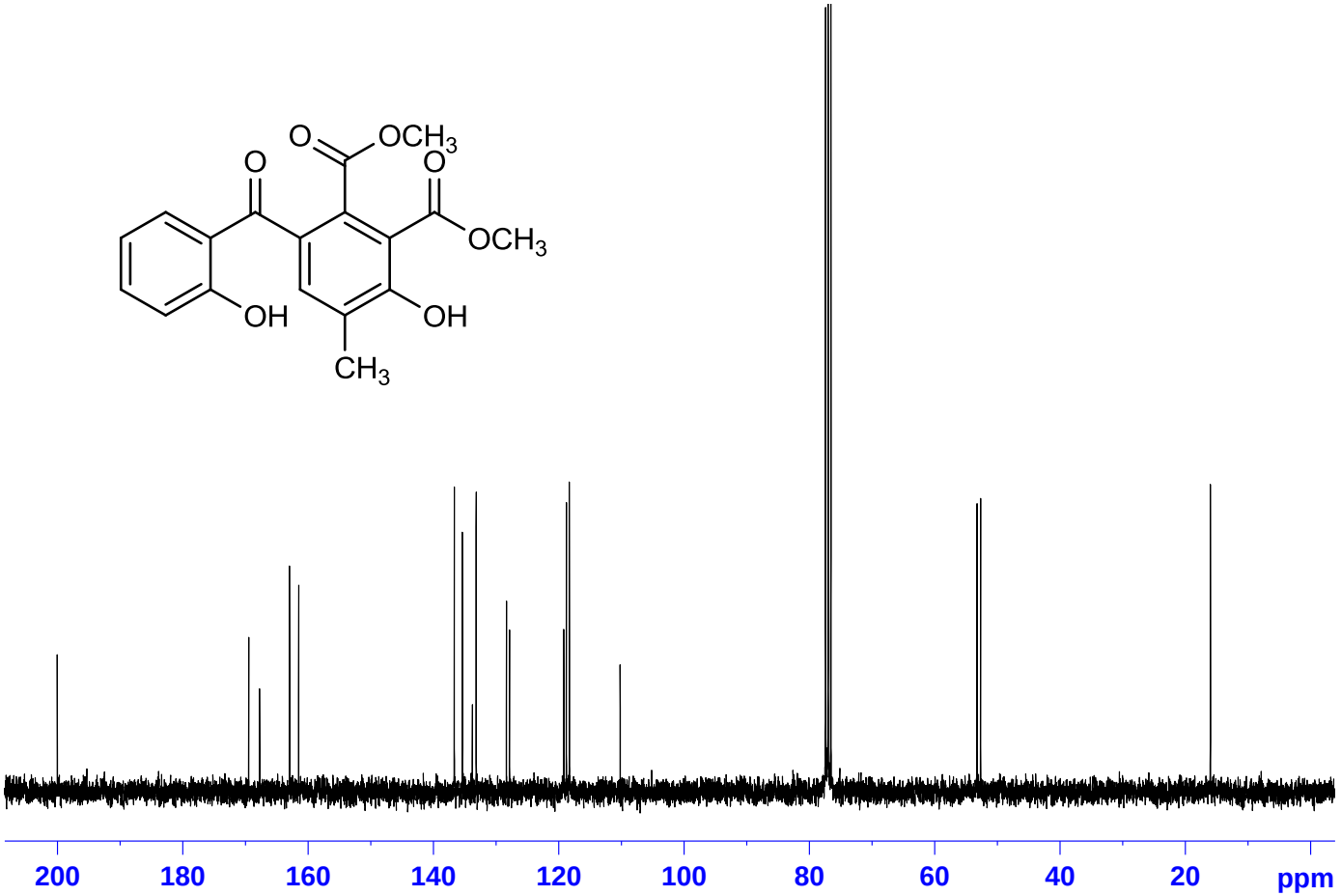
F2 - Acquisition Parameters
Date_ 20100929
Time 7.00
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 4
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 322.5
DW 81.000 usec
DE 6.00 usec
TE 295.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 0.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300153 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

Bunescu/ AB 327
Au13C CDCl3 /opt/topspin 1009 53

200.05
169.49
167.77
162.98
161.53
136.68
135.40
133.80
133.19
128.36
127.87
119.22
118.80
118.33
110.21
53.25
52.68
16.01



Current Data Parameters
NAME AB327 13C
EXPNO 11
PROCNO 1

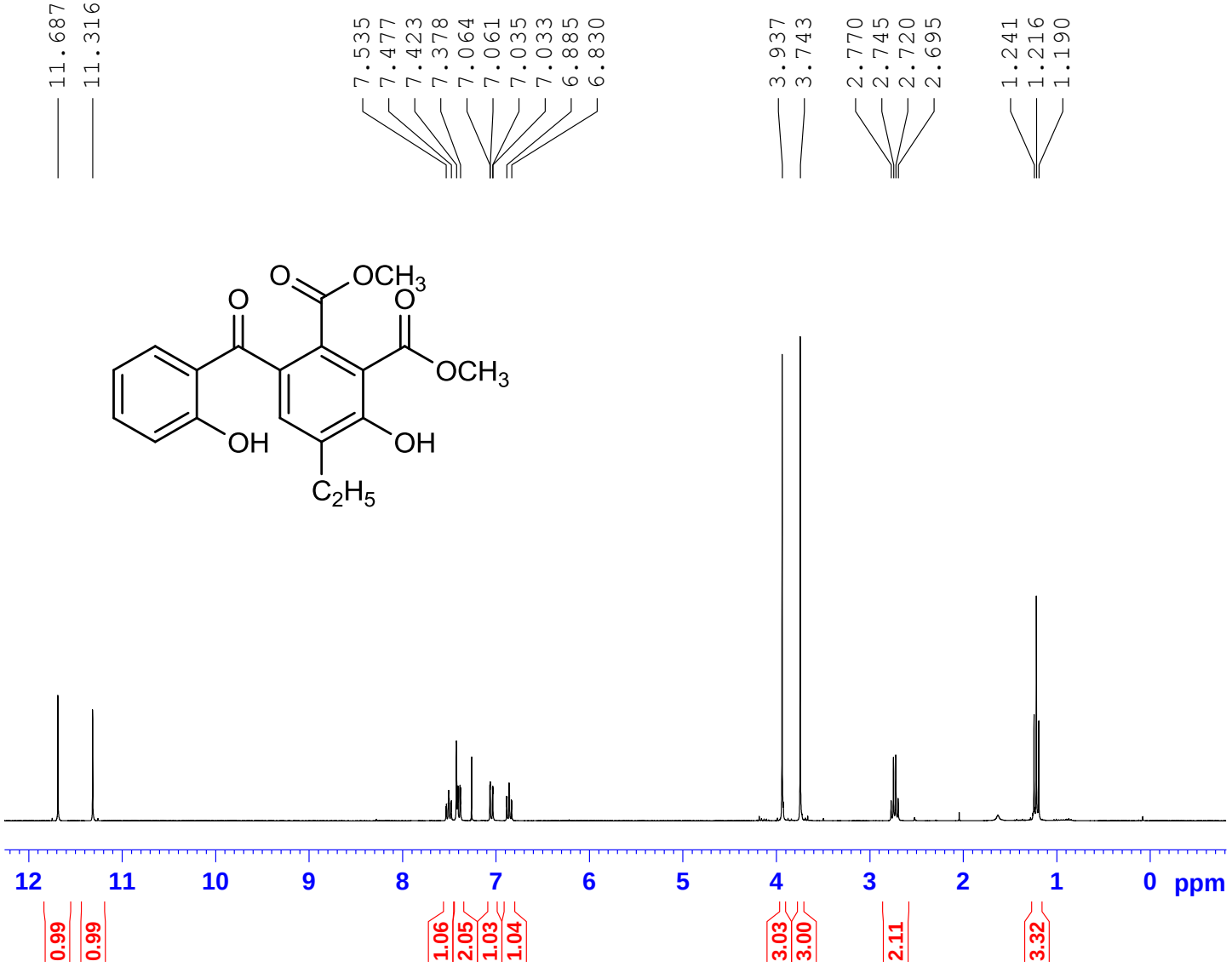
F2 - Acquisition Parameters
Date_ 20100929
Time 7.13
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 256
DS 4
SWH 21097.047 Hz
FIDRES 0.643831 Hz
AQ 0.7766516 sec
RG 32768
DW 23.700 usec
DE 6.00 usec
TE 295.4 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.60 usec
PL1 -1.10 dB
SFO1 75.4771825 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 19.00 dB
PL13 21.00 dB
SFO2 300.1312005 MHz

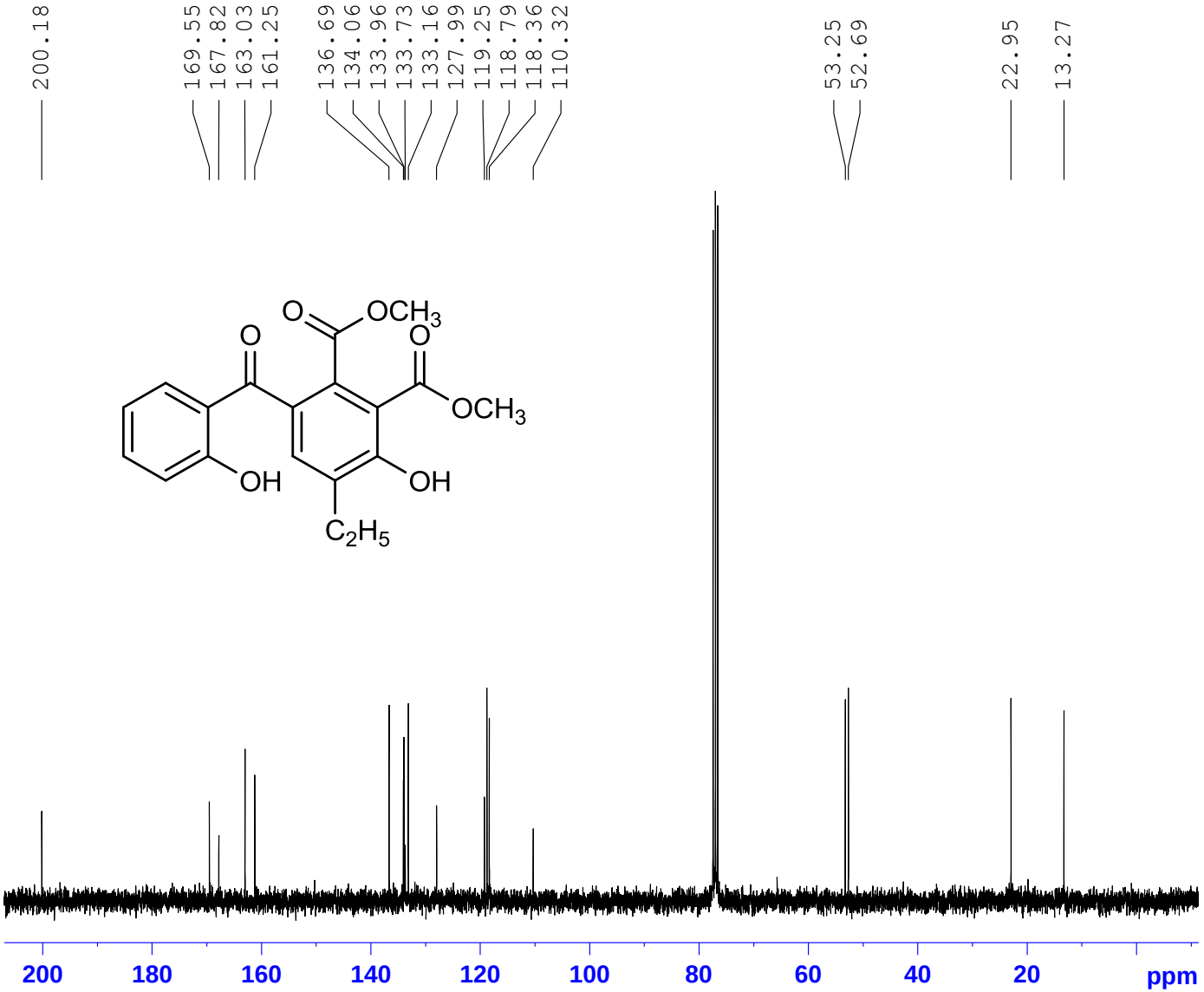
F2 - Processing parameters
SI 32768
SF 75.4677545 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Bunescu/ AB 328
Au1H CDCl3 /opt/topspin 1009 54




```
Current Data Parameters
NAME            AB328 1H
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20100929
Time                7.31
INSTRUM            AV300
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     4
SWH             6172.839 Hz
FIDRES          0.188380 Hz
AQ              2.6542580 sec
RG                 322.5
DW                81.000 usec
DE                 6.00 usec
TE                295.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.00 usec
PL1                 0.00 dB
SFO1         300.1318534 MHz

F2 - Processing parameters
SI                 32768
SF           300.1300153 MHz
WDW                   EM
SSB                    0
LB                  0.10 Hz
GB                     0
PC                  1.00
```

Bunescu/ AB 328
Au13C CDCl3 /opt/topspin 1009 54



Current Data Parameters
NAME AB328 13C
EXPNO 11
PROCNO 1

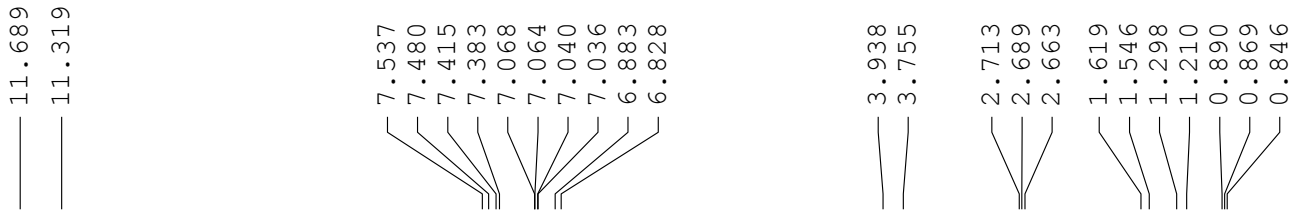
F2 - Acquisition Parameters
Date_ 20100929
Time 7.43
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 256
DS 4
SWH 21097.047 Hz
FIDRES 0.643831 Hz
AQ 0.7766516 sec
RG 32768
DW 23.700 usec
DE 6.00 usec
TE 295.4 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

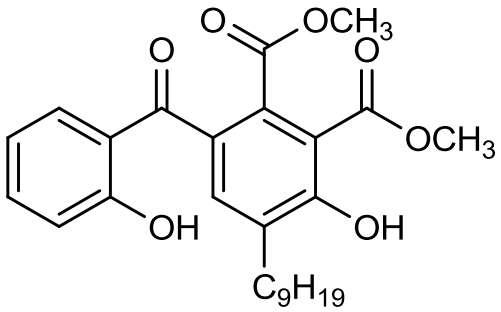
======== CHANNEL f1 ========
NUC1 13C
P1 9.60 usec
PL1 -1.10 dB
SFO1 75.4771825 MHz

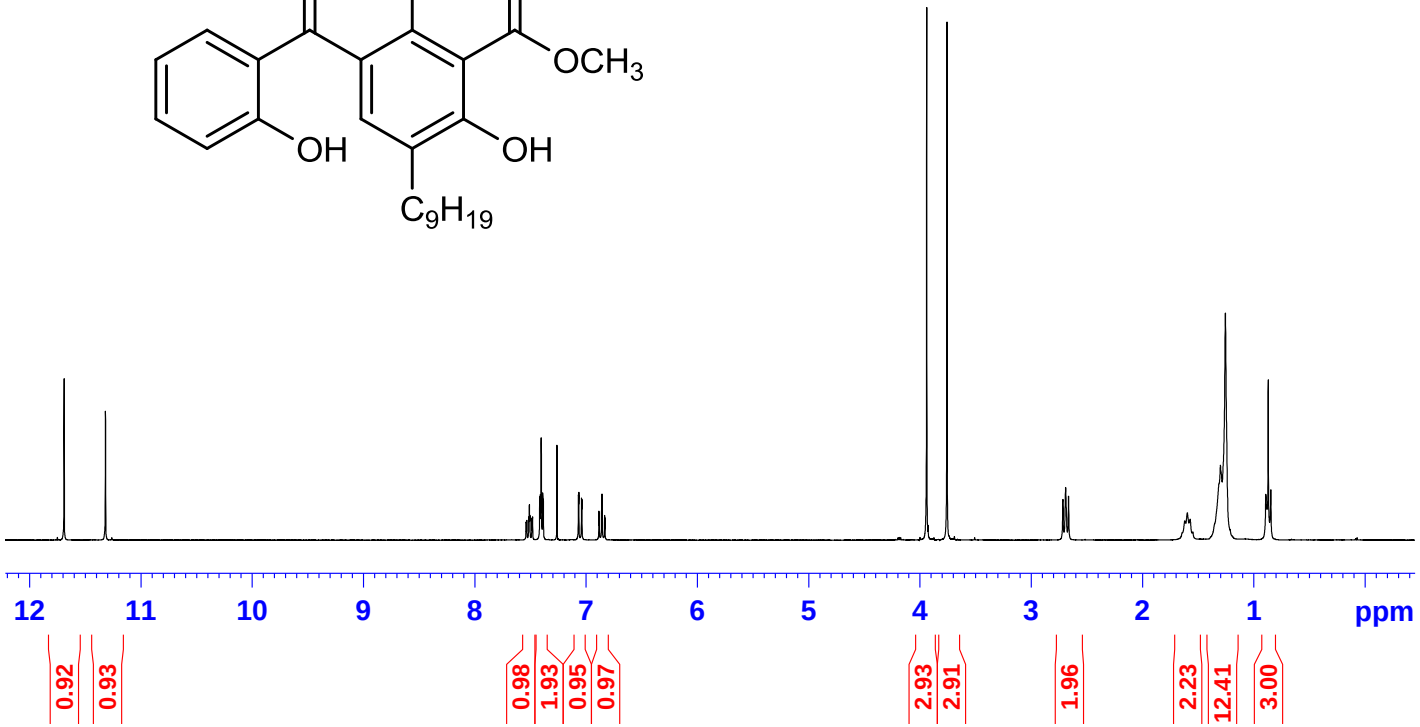
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 19.00 dB
PL13 21.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677540 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Bunescu AB318B
Au1H CDCl3 /opt/topspin 1101 19







Current Data Parameters
NAME AB318
EXPNO 10
PROCNO 1

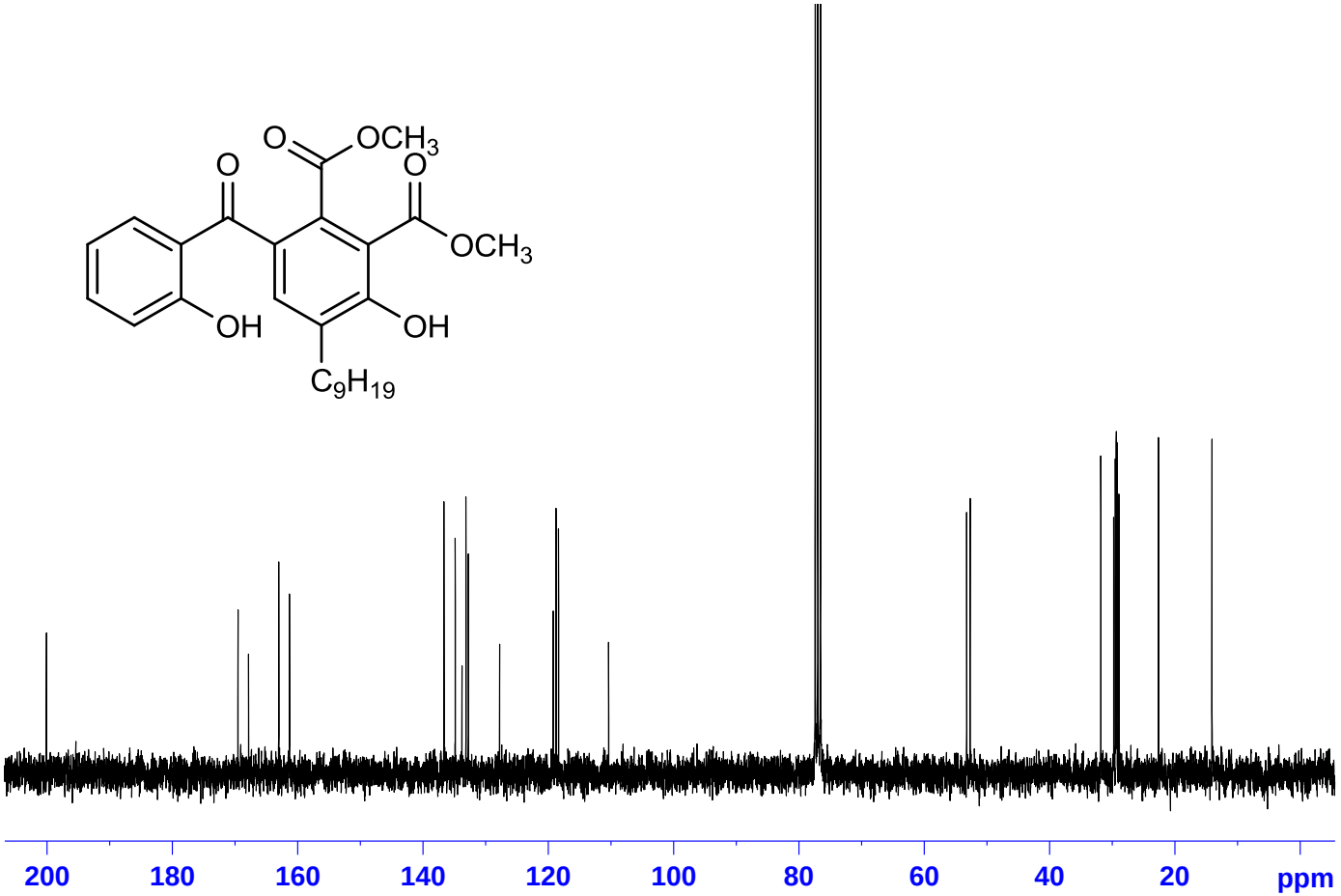
F2 - Acquisition Parameters
Date_ 20110126
Time 21.40
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 4
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 128
DW 81.000 usec
DE 6.00 usec
TE 294.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 0.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300151 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

Bunescu AB318B
Au13C CDCl3 /opt/topspin 1101 19

200.12
169.56
167.87
163.05
161.34
136.68
134.88
133.81
133.17
132.79
127.79
119.25
118.78
118.39
110.40
53.26
52.71
31.84
29.78
29.52
29.40
29.37
29.27
28.95
22.64
14.09



Current Data Parameters
NAME AB318
EXPNO 11
PROCNO 1

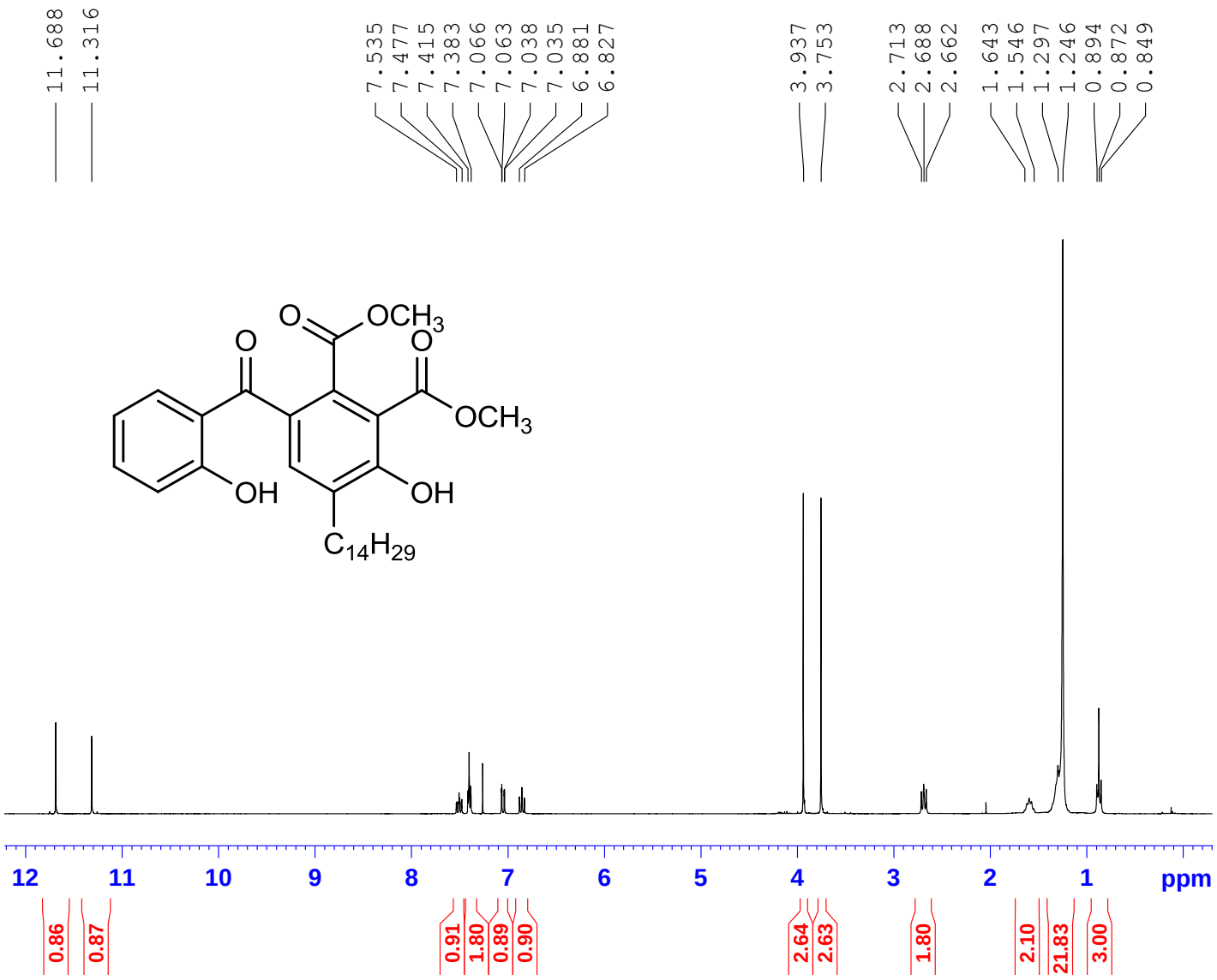
F2 - Acquisition Parameters
Date_ 20110126
Time 21.55
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 256
DS 4
SWH 21097.047 Hz
FIDRES 0.643831 Hz
AQ 0.7766516 sec
RG 32768
DW 23.700 usec
DE 6.00 usec
TE 294.6 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.60 usec
PL1 -1.10 dB
SFO1 75.4771825 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 19.00 dB
PL13 21.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677537 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Bunescu / AB340
Au1H CDCl3 /opt/topspin 1010 24



Current Data Parameters
NAME AB340
EXPNO 10
PROCNO 1

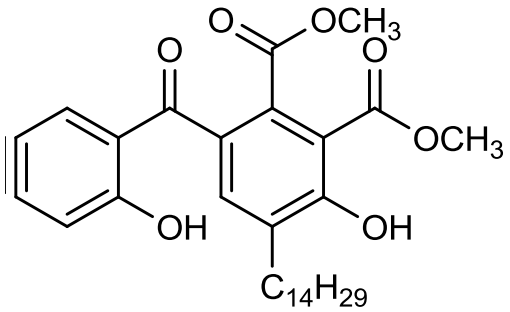
F2 - Acquisition Parameters
Date_ 20101019
Time 21.15
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 4
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 101.6
DW 81.000 usec
DE 6.00 usec
TE 294.9 K
D1 1.00000000 sec
TD0 1

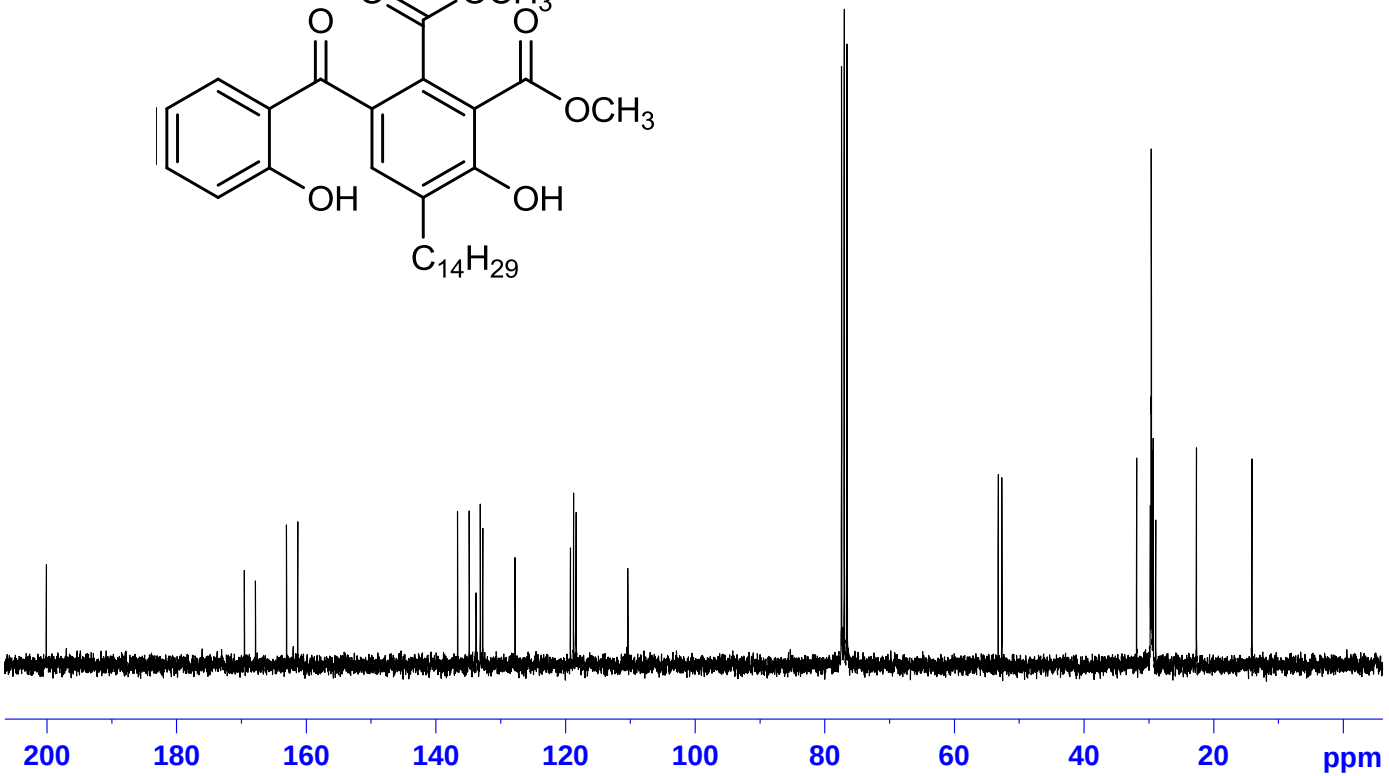
======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 0.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300153 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

Bunescu / AB340
Au13C CDCl3 /opt/topspin 1010 24

200.12
169.56
167.85
163.05
161.34
136.67
134.86
133.80
133.16
132.78
127.79
119.25
118.76
118.38
110.40
53.25
52.69
31.90
29.78
29.67
29.63
29.57
29.40
29.38
29.33
28.95
22.67
14.10





Current Data Parameters
NAME AB340
EXPNO 11
PROCNO 1

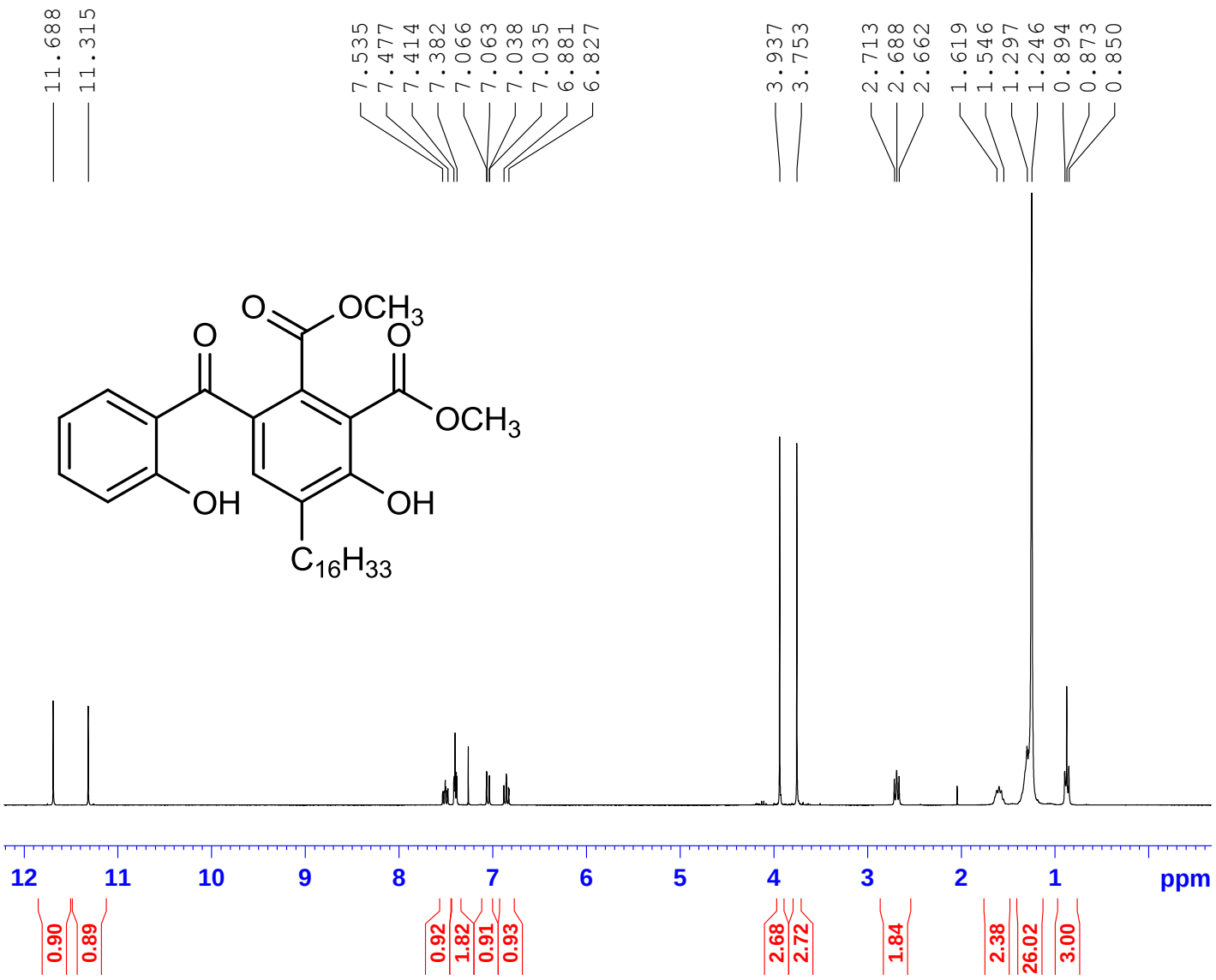
F2 - Acquisition Parameters
Date_ 20101019
Time 21.30
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 256
DS 4
SWH 21097.047 Hz
FIDRES 0.643831 Hz
AQ 0.7766516 sec
RG 32768
DW 23.700 usec
DE 6.00 usec
TE 295.3 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.60 usec
PL1 -1.10 dB
SFO1 75.4771825 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 19.00 dB
PL13 21.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677541 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

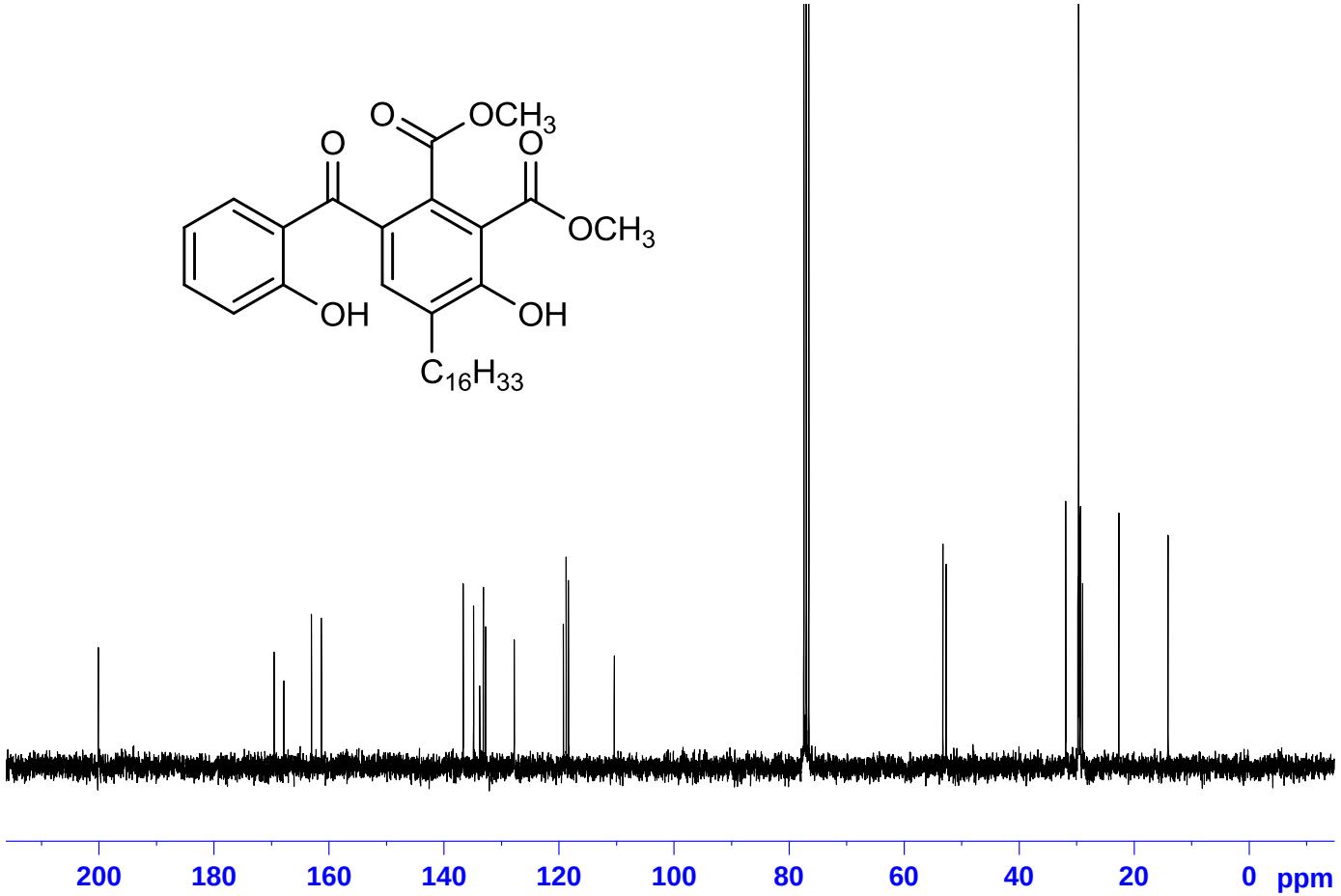
Bunescu/ AB 321
Au1H CDCl3 /opt/topspin 1009 24




```
Current Data Parameters
NAME                  AB321 1H
EXPNO                       10
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20100921
Time                     20.56
INSTRUM                  AV300
PROBHD    5 mm PABBO BB-
PULPROG                  zg30
TD                      32768
SOLVENT                 CDCl3
NS                         32
DS                          4
SWH                 6172.839 Hz
FIDRES              0.188380 Hz
AQ                  2.6542580 sec
RG                        114
DW                   81.000 usec
DE                    6.00 usec
TE                    294.9 K
D1                1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                    11.00 usec
PL1                    0.00 dB
SFO1            300.1318534 MHz

F2 - Processing parameters
SI                      32768
SF              300.1300153 MHz
WDW                        EM
SSB                         0
LB                    0.10 Hz
GB                          0
PC                     1.00
```

Bunescu/ AB 321
Au13C CDCl3 /opt/topspin 1009 24

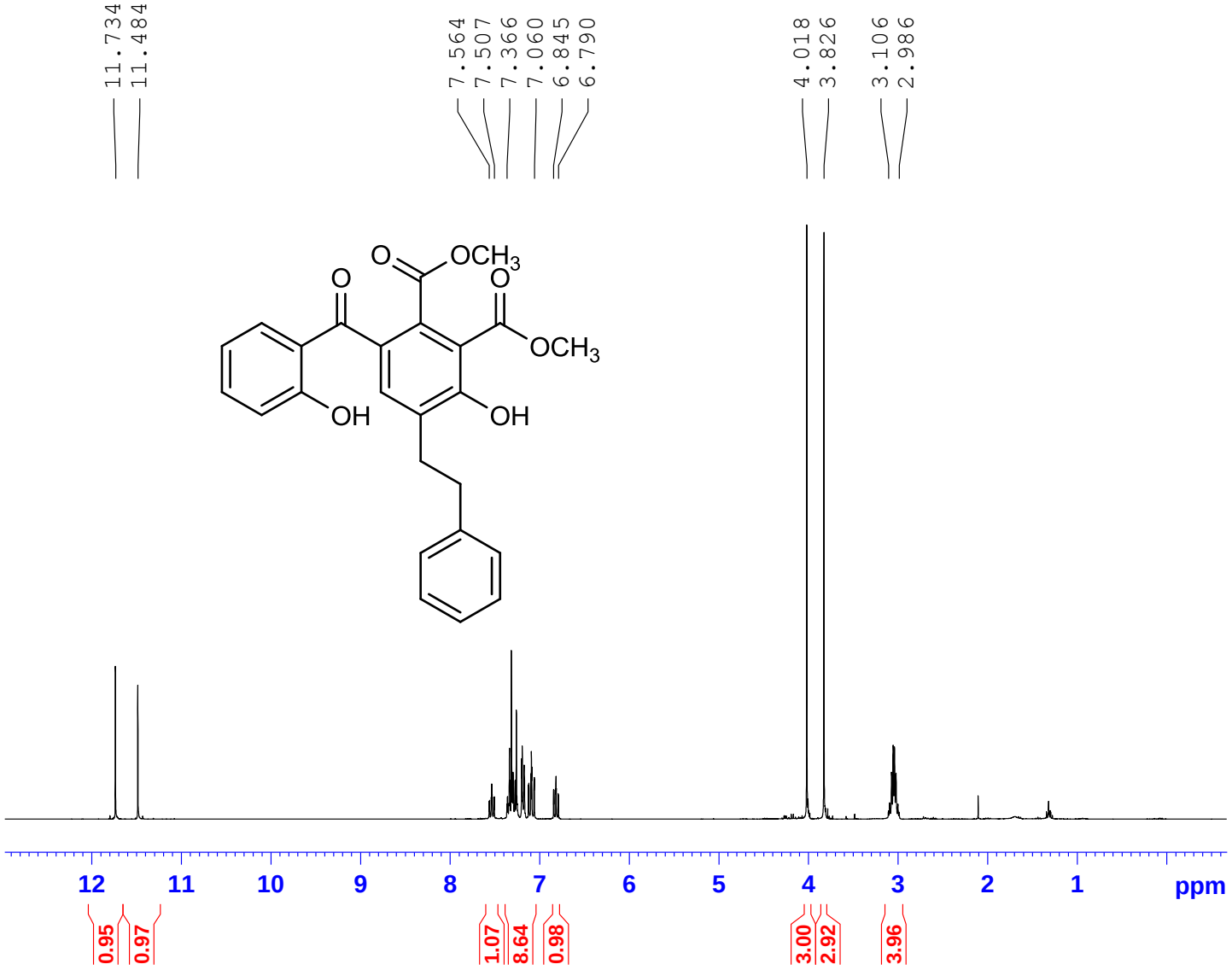
200.12
169.56
167.87
163.06
161.34
136.67
134.86
133.81
133.17
132.79
127.80
119.26
118.77
118.38
110.40
53.25
52.70
31.90
29.78
29.68
29.64
29.58
29.41
29.39
29.34
28.96
22.67
14.11



BRUKER

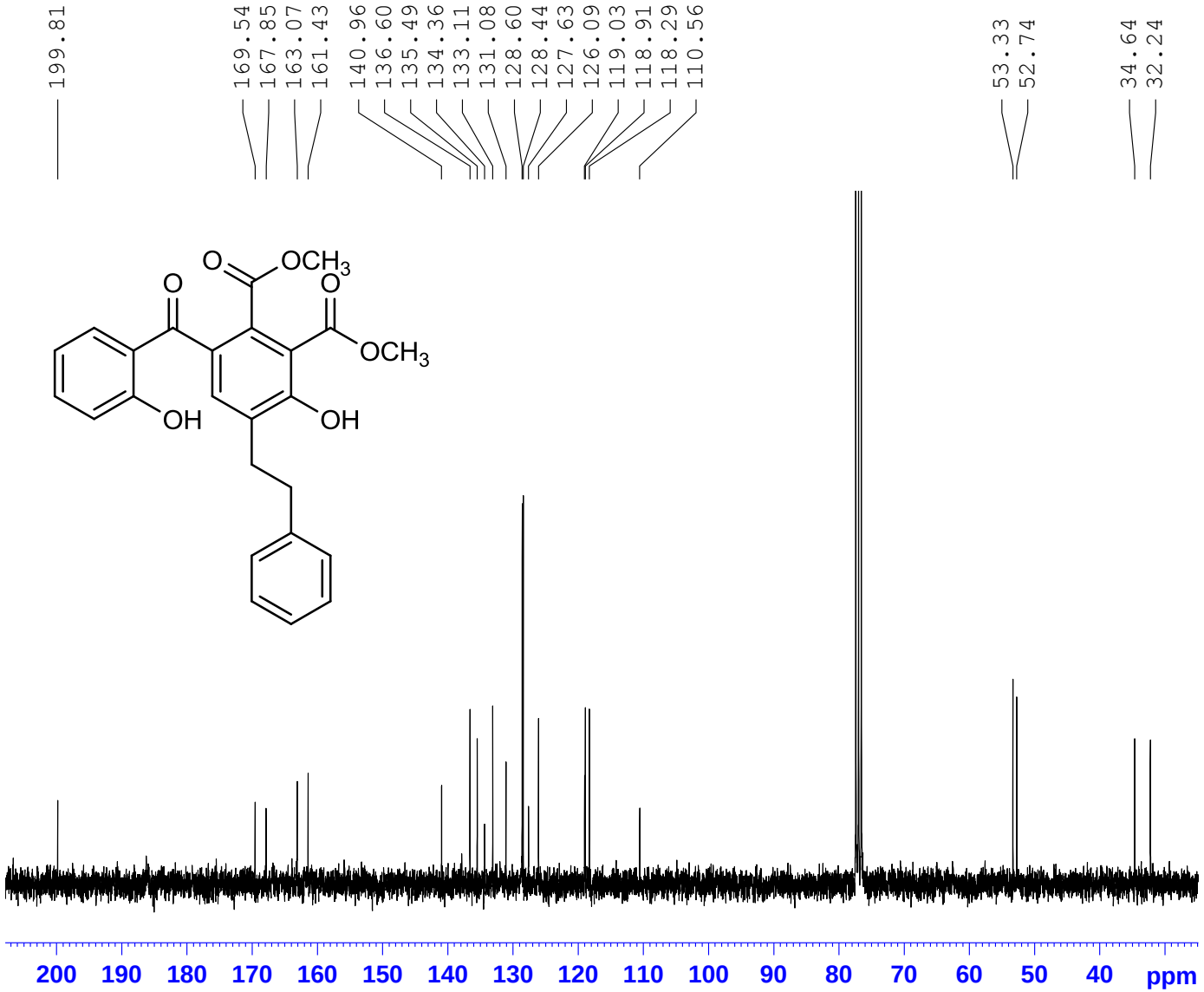
```
Current Data Parameters
NAME                  AB321 13C
EXPNO                        11
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20100921
Time                      21.09
INSTRUM                   AV300
PROBHD    5 mm PABBO BB-
PULPROG                  zgpg30
TD                        32768
SOLVENT                   CDCl3
NS                          256
DS                            4
SWH                  21097.047 Hz
FIDRES                0.643831 Hz
AQ                    0.7766516 sec
RG                        32768
DW                       23.700 usec
DE                        6.00 usec
TE                       295.4 K
D1                    2.00000000 sec
d11                   0.03000000 sec
DELTA                 1.89999998 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                        13C
P1                        9.60 usec
PL1                      -1.10 dB
SFO1                75.4771825 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                   100.00 usec
PL2                       0.00 dB
PL12                     19.00 dB
PL13                     21.00 dB
SFO2                300.1312005 MHz

F2 - Processing parameters
SI                        32768
SF                  75.4677536 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40
```

Bumena AB 302 A
Au1H CDCl3 /opt/topspin 1010 32




```
Current Data Parameters
NAME          AB302A 1H
EXPNO                10
PROCNO                1

F2 - Acquisition Parameters
Date_          20101009
Time               9.20
INSTRUM           AV300
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   32
DS                    4
SWH            6172.839 Hz
FIDRES         0.188380 Hz
AQ             2.6542580 sec
RG                 322.5
DW                81.000 usec
DE                 6.00 usec
TE                294.7 K
D1             1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                11.00 usec
PL1                0.00 dB
SFO1         300.1318534 MHz

F2 - Processing parameters
SI                32768
SF           300.1299984 MHz
WDW                  EM
SSB                   0
LB                 0.10 Hz
GB                    0
PC                 1.00
```

Bumena AB 302 A
Au13C CDCl3 /opt/topspin 1010 32



Current Data Parameters
NAME AB302 13C
EXPNO 11
PROCNO 1

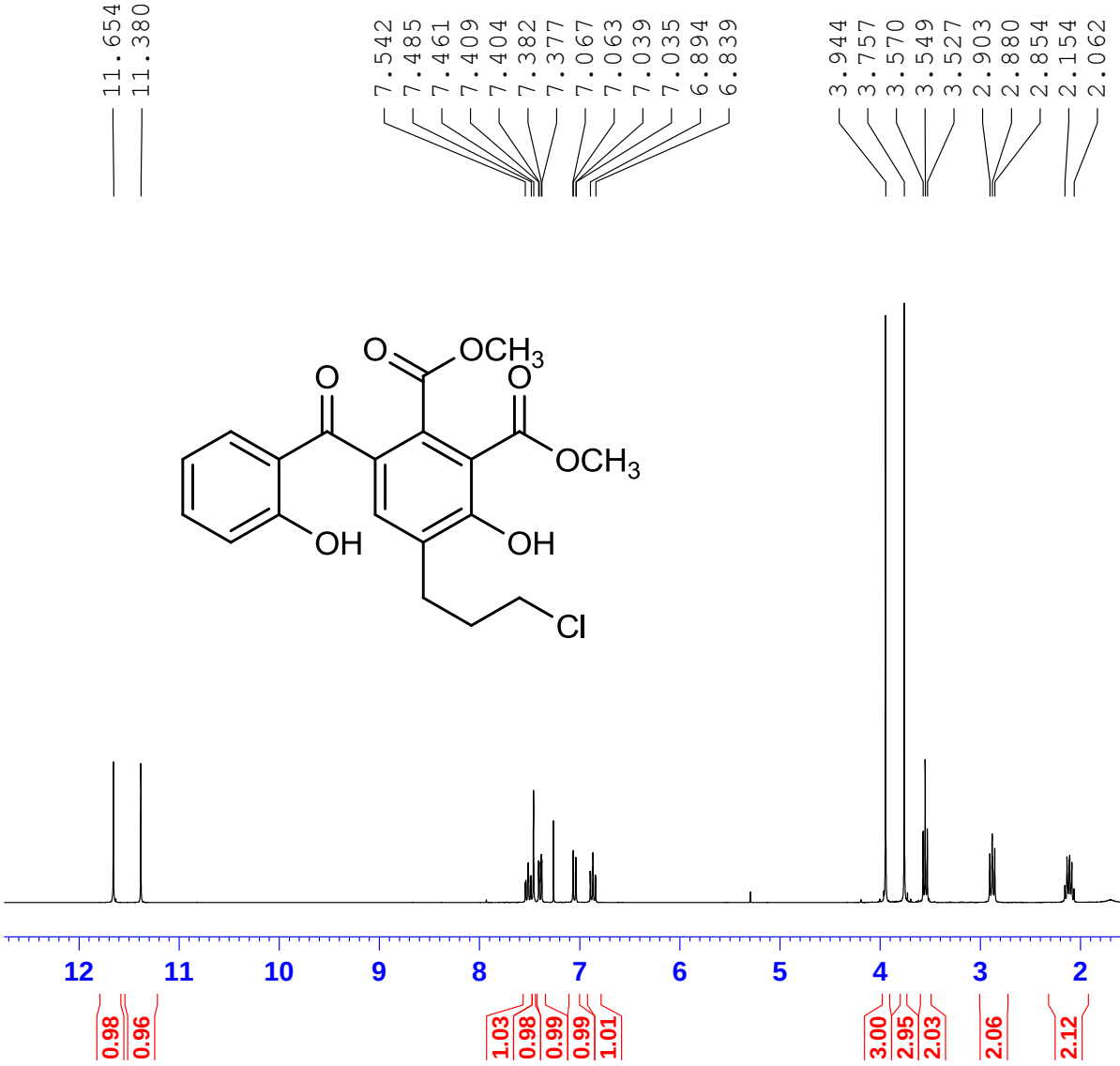
F2 - Acquisition Parameters
Date_ 20101009
Time 9.33
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 256
DS 4
SWH 21097.047 Hz
FIDRES 0.643831 Hz
AQ 0.7766516 sec
RG 32768
DW 23.700 usec
DE 6.00 usec
TE 295.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.60 usec
PL1 -1.10 dB
SFO1 75.4771825 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 19.00 dB
PL13 21.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677537 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

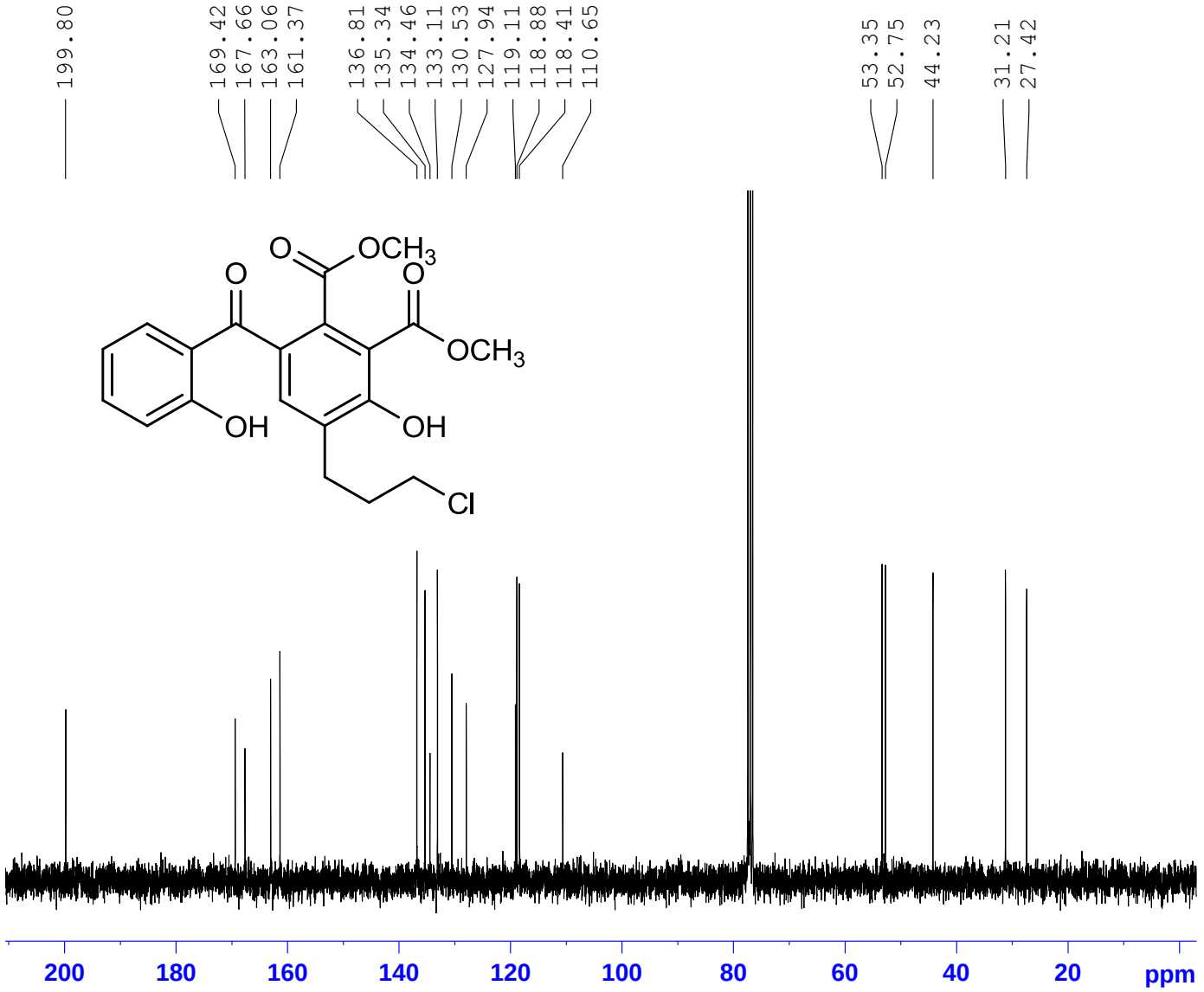
Bunescu/ AB 317
Au1H CDCl3 /opt/topspin 1009 28



BRUKER

```
Current Data Parameters
NAME            AB317 1H
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20100908
Time                9.21
INSTRUM            AV300
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     4
SWH             6172.839 Hz
FIDRES          0.188380 Hz
AQ              2.6542580 sec
RG                 322.5
DW                81.000 usec
DE                 6.00 usec
TE                295.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.00 usec
PL1                 0.00 dB
SFO1         300.1318534 MHz

F2 - Processing parameters
SI                 32768
SF           300.1300153 MHz
WDW                   EM
SSB                    0
LB                  0.10 Hz
GB                     0
PC                  1.00
```

Bunescu/ AB 317
Au13C CDCl3 /opt/topspin 1009 28



Current Data Parameters
NAME AB317 13C
EXPNO 11
PROCNO 1

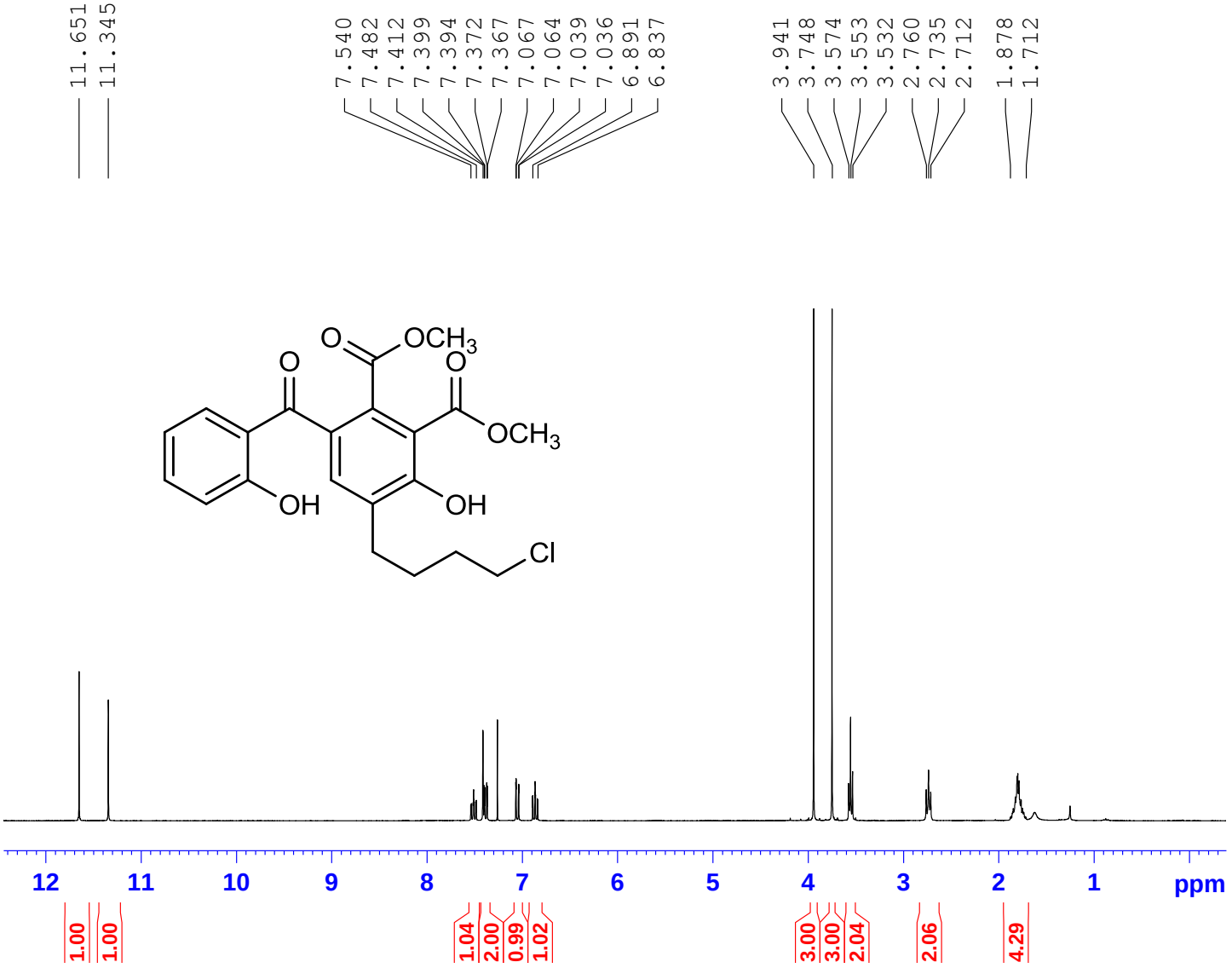
F2 - Acquisition Parameters
Date_ 20100908
Time 9.34
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 256
DS 4
SWH 21097.047 Hz
FIDRES 0.643831 Hz
AQ 0.7766516 sec
RG 32768
DW 23.700 usec
DE 6.00 usec
TE 295.5 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.60 usec
PL1 -1.10 dB
SFO1 75.4771825 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 19.00 dB
PL13 21.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677548 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Bunescu/ AB 296 B
Au1H CDCl3 /opt/topspin 1008 33



Current Data Parameters
NAME AB296B 1H
EXPNO 10
PROCNO 1

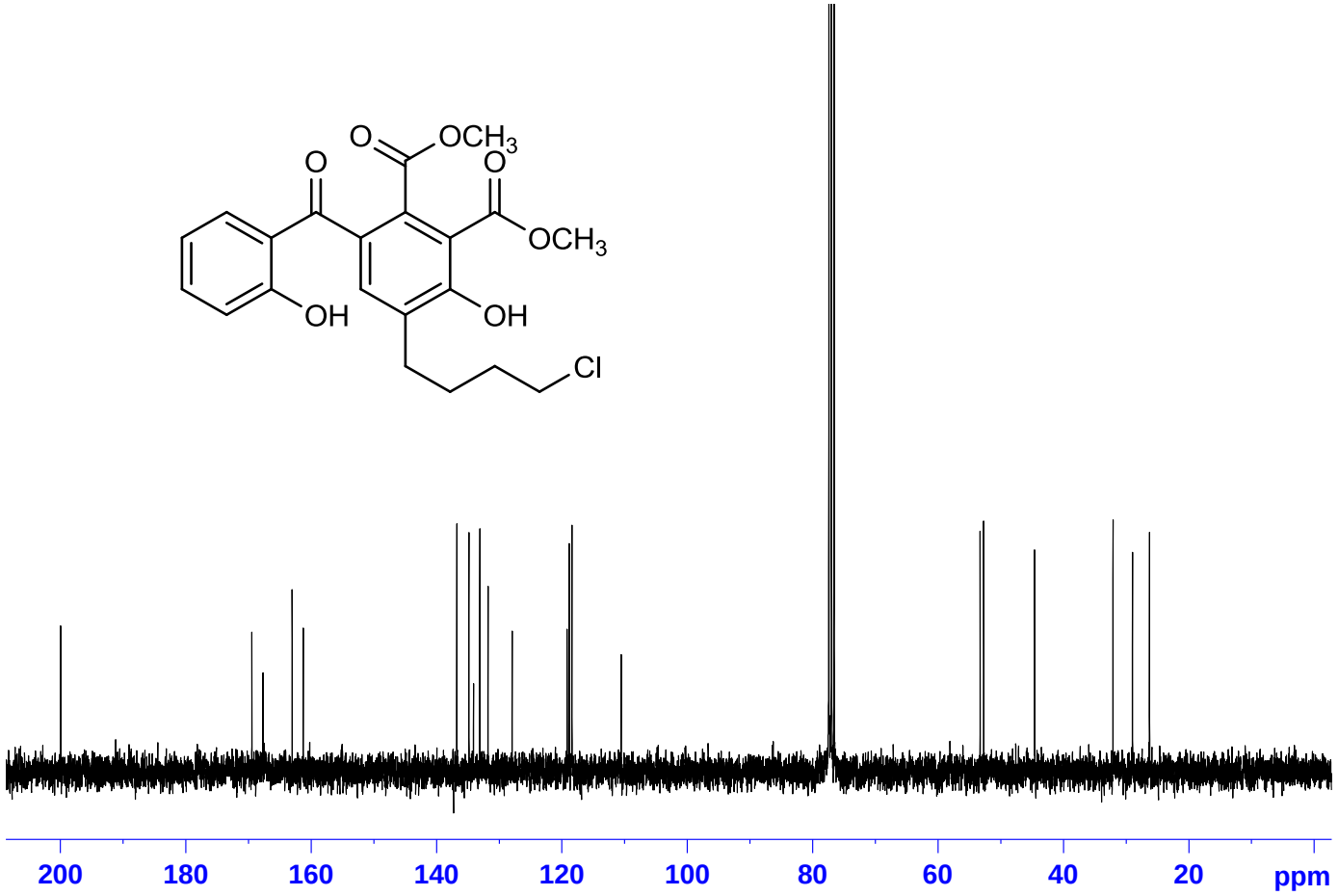
F2 - Acquisition Parameters
Date_ 20100809
Time 23.55
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 4
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 322.5
DW 81.000 usec
DE 6.00 usec
TE 296.4 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 0.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300154 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

Bunescu/ AB 296 B
Au13C CDCl3 /opt/topspin 1008 33

199.97
169.49
167.71
163.04
161.27
136.77
134.86
134.11
133.11
131.80
127.95
119.20
118.85
118.41
110.55
53.32
52.73
44.60
32.10
28.99
26.27



Current Data Parameters
NAME AB296B 13C
EXPNO 11
PROCNO 1

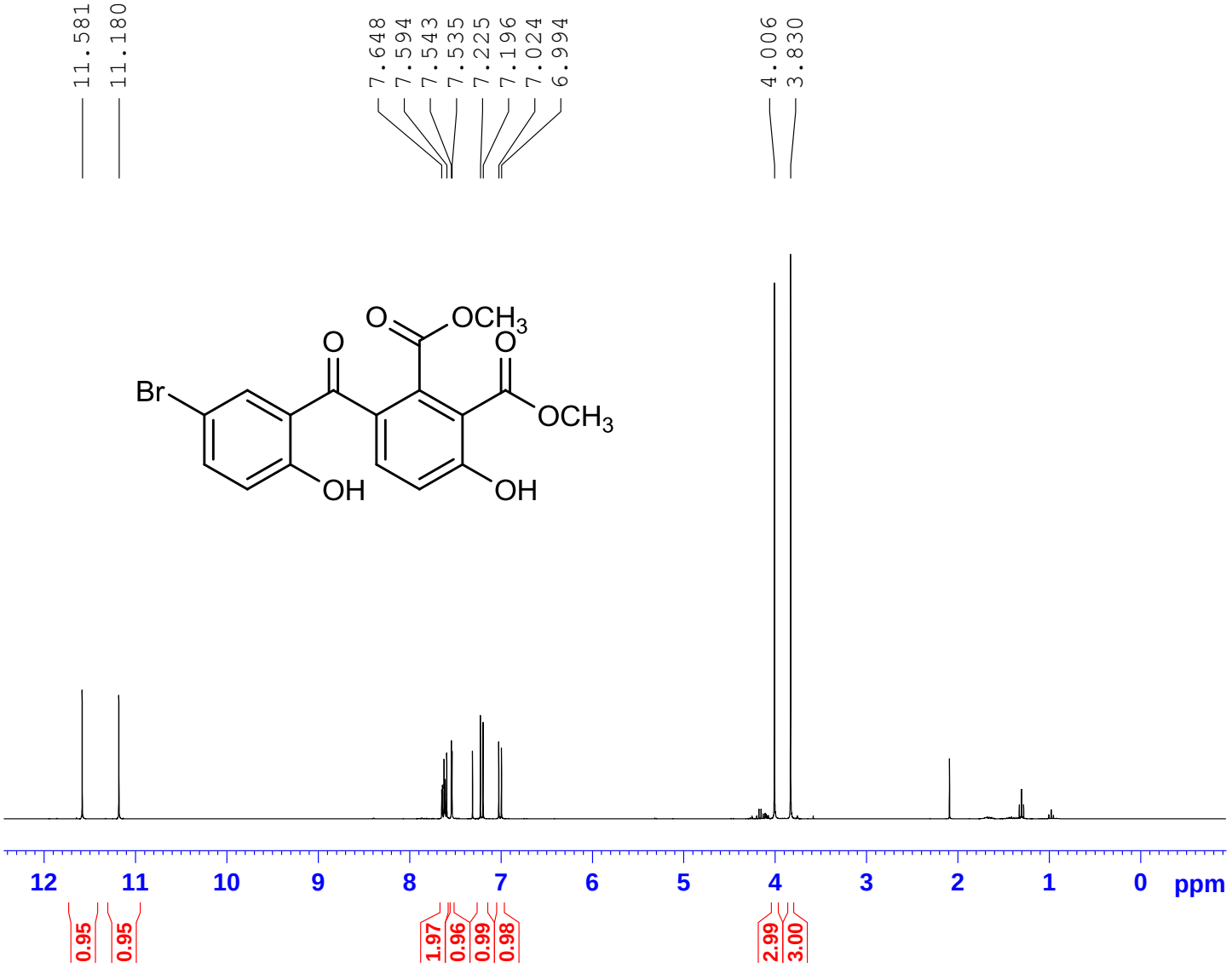
F2 - Acquisition Parameters
Date_ 20100810
Time 0.08
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 256
DS 4
SWH 21097.047 Hz
FIDRES 0.643831 Hz
AQ 0.7766516 sec
RG 32768
DW 23.700 usec
DE 6.00 usec
TE 296.8 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.60 usec
PL1 -1.10 dB
SFO1 75.4771825 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 19.00 dB
PL13 21.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677539 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Bunescu/ AB 383
Au1H CDCl3 /opt/topspin 1103 26



Current Data Parameters
NAME AB383
EXPNO 10
PROCNO 1

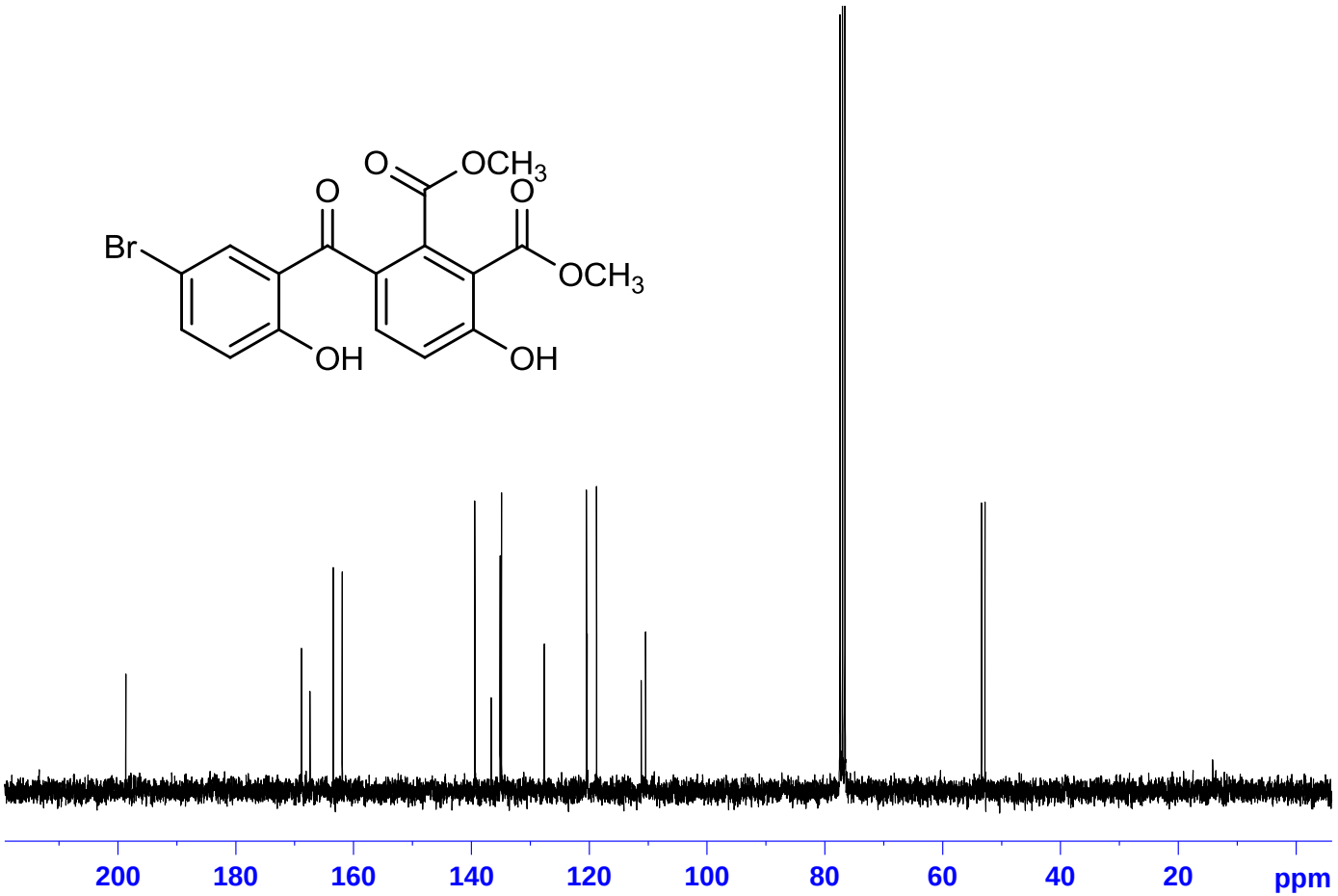
F2 - Acquisition Parameters
Date_ 20110303
Time 0.27
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 4
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 322.5
DW 81.000 usec
DE 6.00 usec
TE 294.6 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 0.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

Bunescu/ AB 383
Au13C CDCl3 /opt/topspin 1103 12

198.68
168.85
167.42
163.45
161.94
139.43
136.64
135.13
134.90
127.64
120.49
120.37
118.76
111.19
110.43
53.42
52.83



Current Data Parameters
NAME AB383 13C
EXPNO 10
PROCNO 1

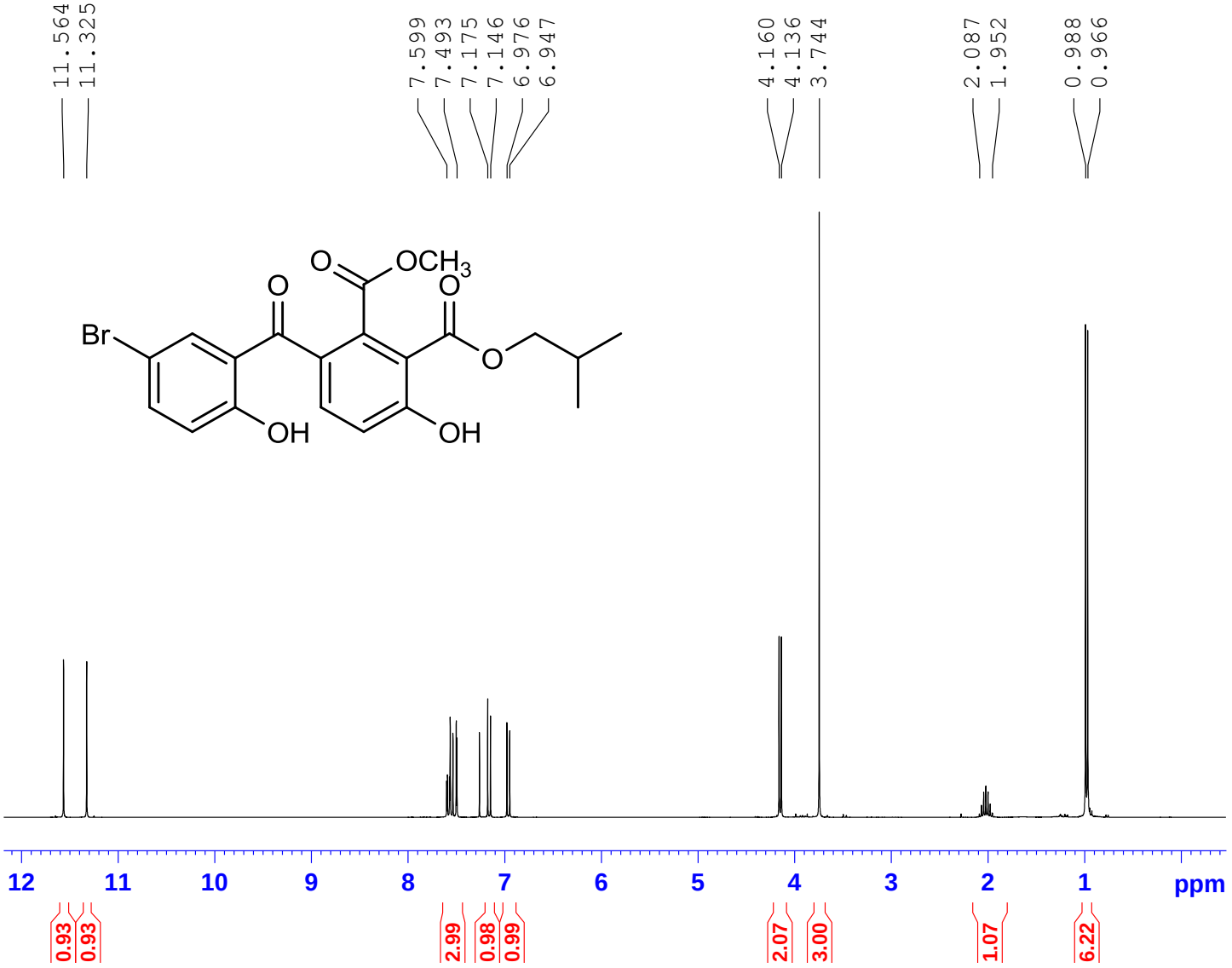
F2 - Acquisition Parameters
Date_ 20110303
Time 19.29
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 256
DS 4
SWH 21097.047 Hz
FIDRES 0.643831 Hz
AQ 0.7766516 sec
RG 32768
DW 23.700 usec
DE 6.00 usec
TE 295.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.60 usec
PL1 -1.10 dB
SFO1 75.4771825 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 19.00 dB
PL13 21.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677542 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Bunescu/ AB373 B
Au1H CDCl3 /opt/topspin 1102 49



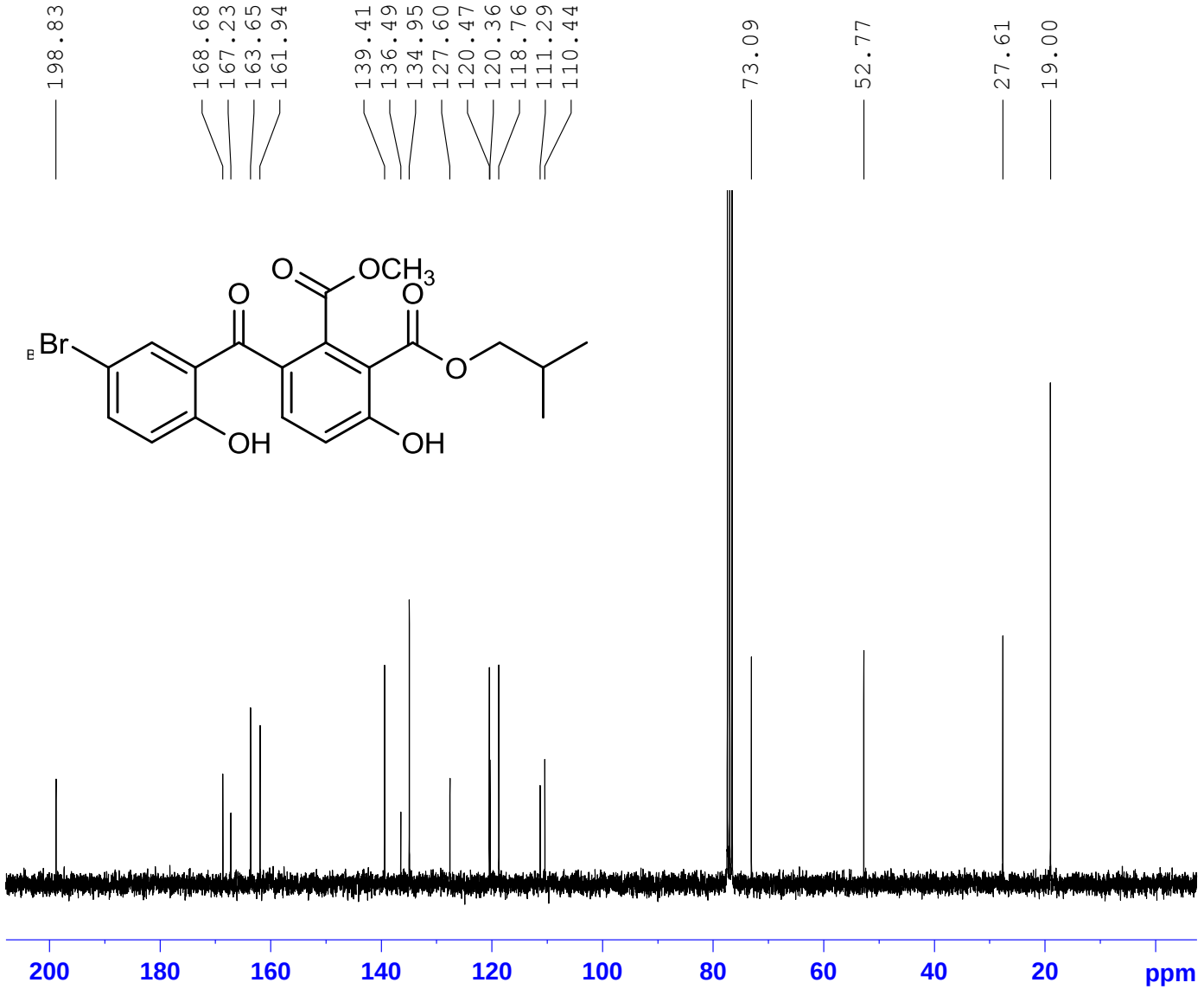
Current Data Parameters
NAME AB373B
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110217
Time 17.11
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 4
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 322.5
DW 81.000 usec
DE 6.00 usec
TE 294.2 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 0.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300152 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

Bunescu/ AB373 B
Au13C CDCl3 /opt/topspin 1102 49



Current Data Parameters
NAME AB373B
EXPNO 11
PROCNO 1

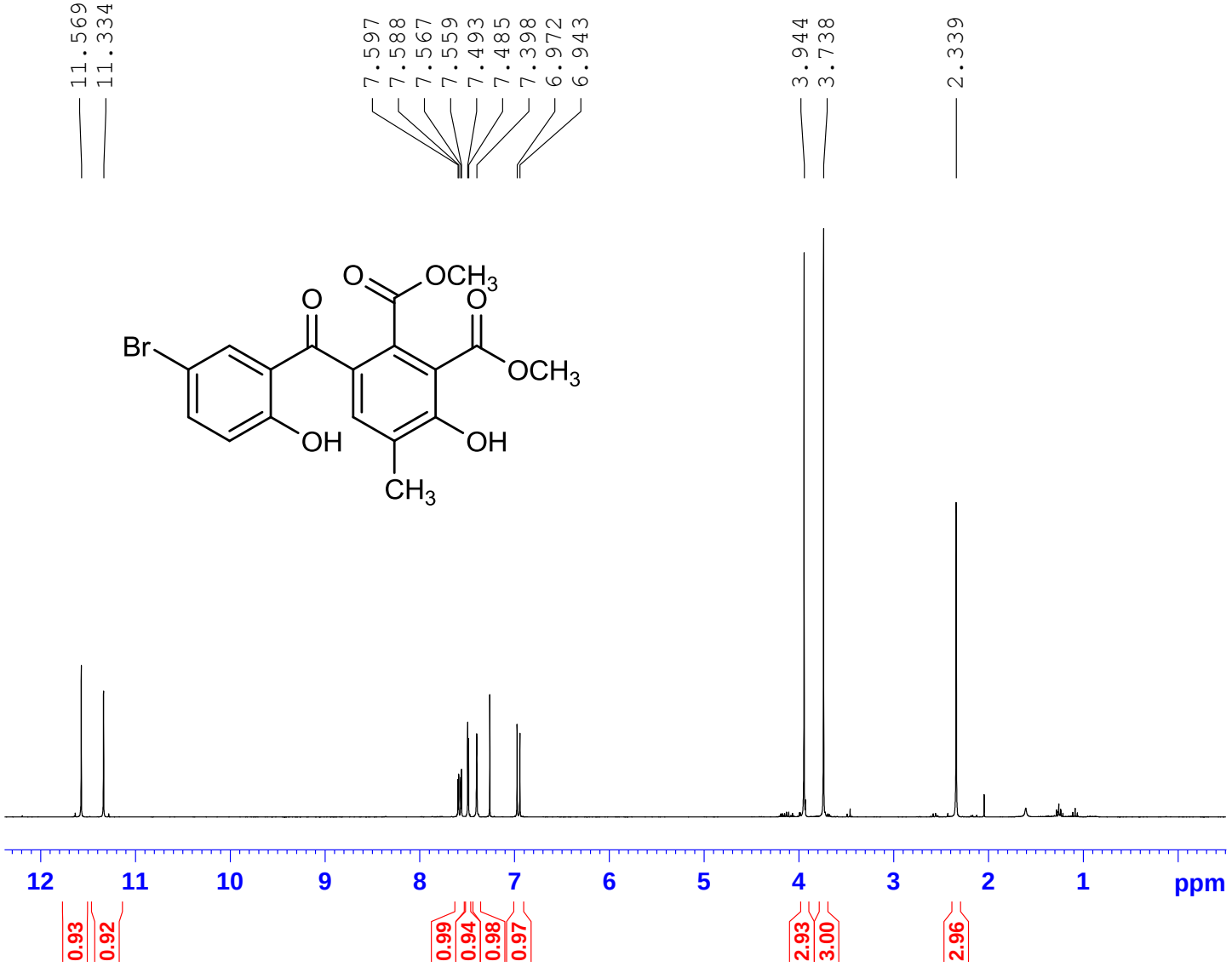
F2 - Acquisition Parameters
Date_ 20110217
Time 17.24
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 256
DS 4
SWH 21097.047 Hz
FIDRES 0.643831 Hz
AQ 0.7766516 sec
RG 32768
DW 23.700 usec
DE 6.00 usec
TE 294.7 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.60 usec
PL1 -1.10 dB
SFO1 75.4771825 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 19.00 dB
PL13 21.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677542 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Bunsescu AB371
Au1H CDCl3 /opt/topspin 1101 21



Current Data Parameters

NAME	AB371
EXPNO	10
PROCNO	1

F2 - Acquisition Parameters

Date_	20110124
Time	19.34
INSTRUM	AV300
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	32
DS	4
SWH	6172.839 Hz
FIDRES	0.188380 Hz
AQ	2.6542580 sec
RG	322.5
DW	81.000 usec
DE	6.00 usec
TE	294.1 K
D1	1.00000000 sec
TD0	1

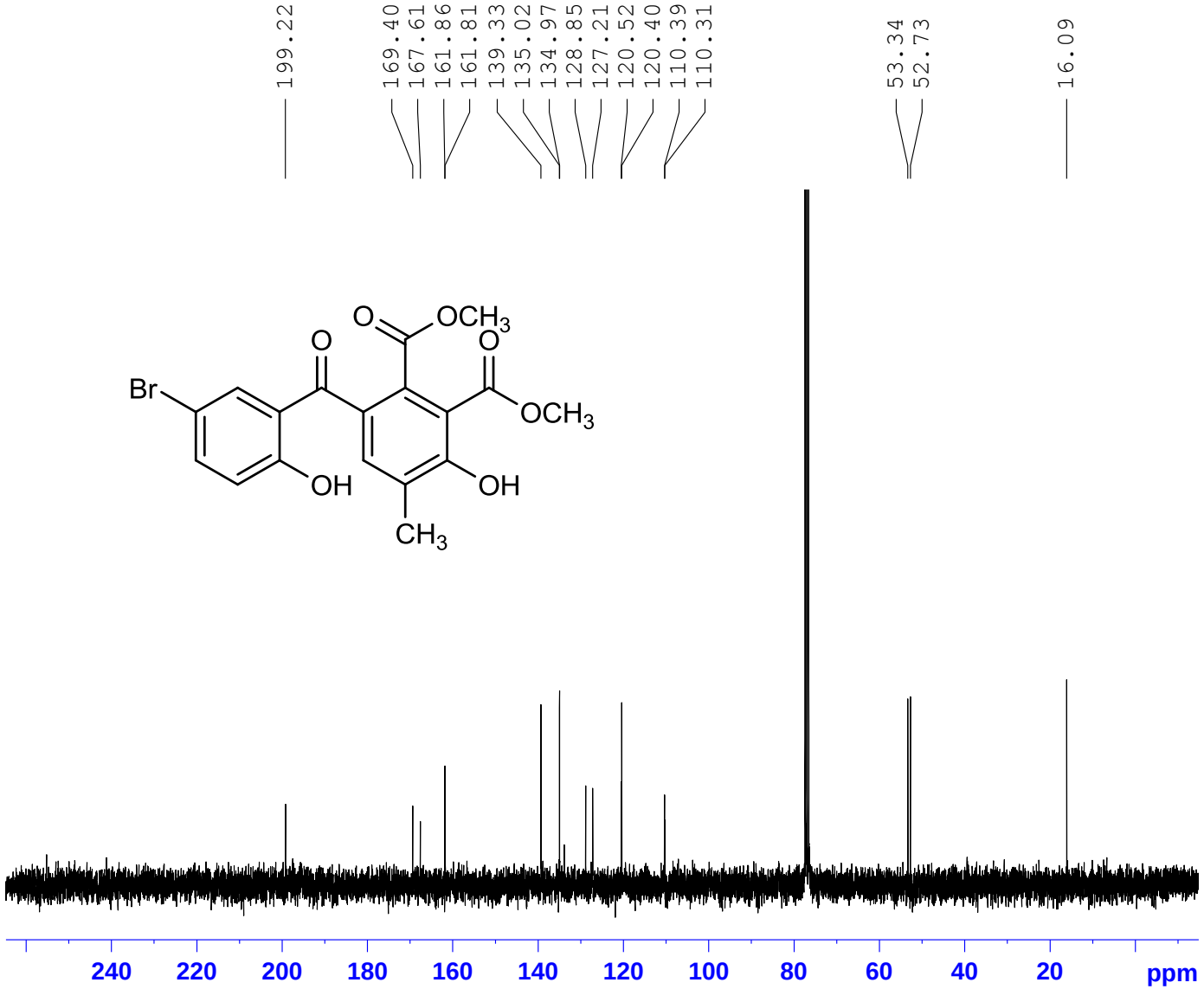
======== CHANNEL f1 ========

NUC1	1H
P1	11.00 usec
PL1	0.00 dB
SFO1	300.1318534 MHz

F2 - Processing parameters

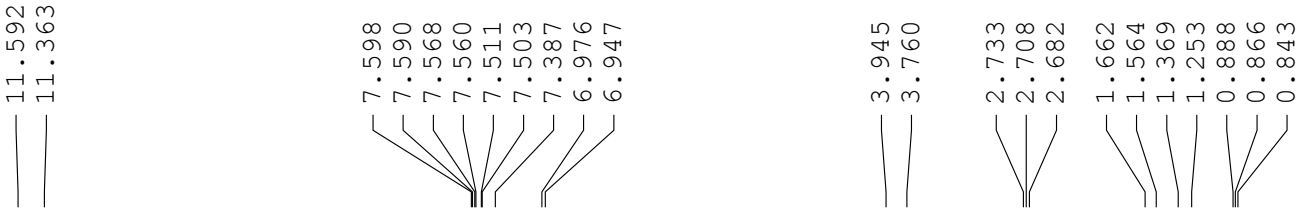
SI	32768
SF	300.1300152 MHz
WDW	EM
SSB	0
LB	0.10 Hz
GB	0
PC	1.00

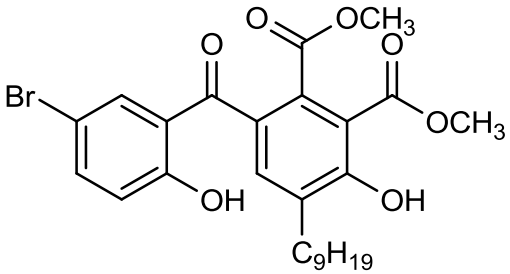
Bunsescu AB371
Au13C CDCl3 /opt/topspin 1101 21

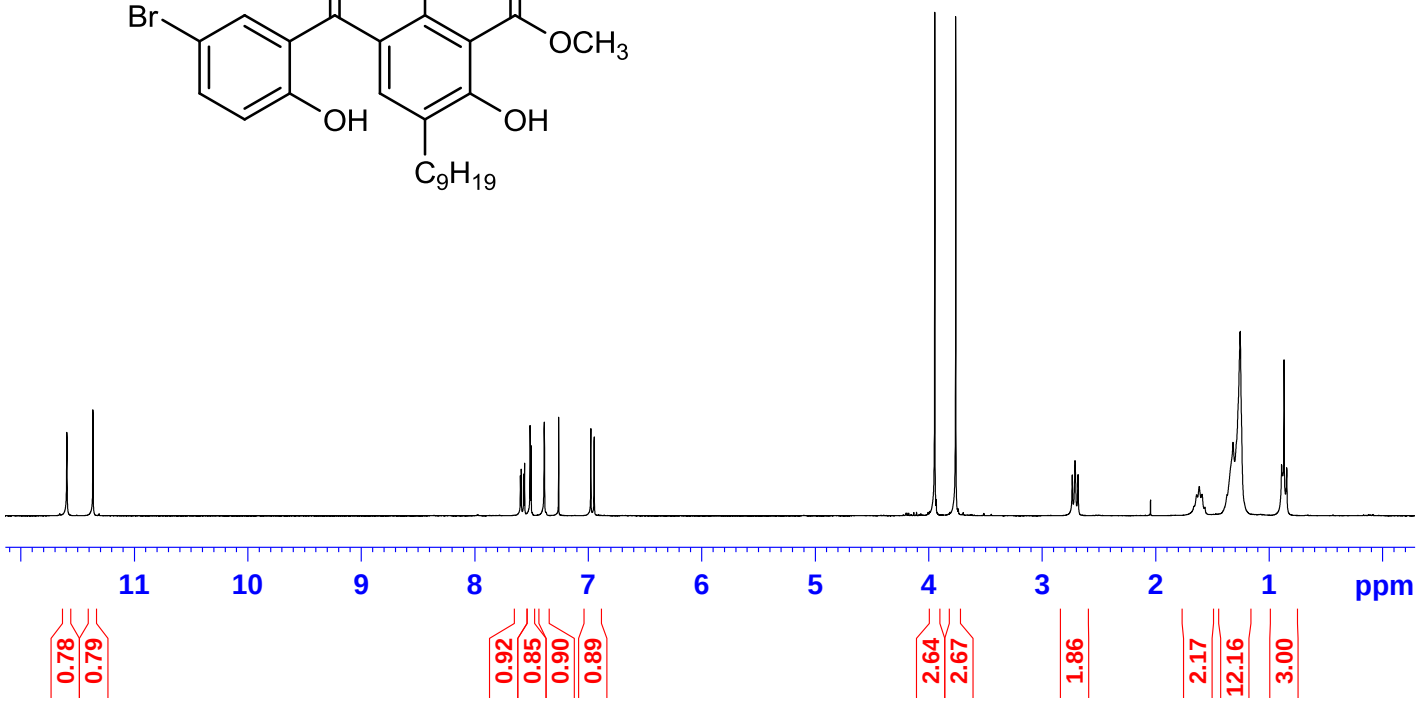



```
Current Data Parameters
NAME                AB371
EXPNO                  11
PROCNO                  1

F2 - Acquisition Parameters
Date_            20110124
Time                19.48
INSTRUM             AV300
PROBHD    5 mm PABBO BB-
PULPROG           zgpg30
TD                 32768
SOLVENT            CDCl3
NS                   256
DS                     4
SWH            21097.047 Hz
FIDRES          0.643831 Hz
AQ              0.7766516 sec
RG                 32768
DW                23.700 usec
DE                 6.00 usec
TE                294.6 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                  9.60 usec
PL1                -1.10 dB
SFO1          75.4771825 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2                 0.00 dB
PL12               19.00 dB
PL13               21.00 dB
SFO2         300.1312005 MHz

F2 - Processing parameters
SI                 32768
SF            75.4677534 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

Bunescu AB374
Au1H CDCl3 /opt/topspin 1102 7







Current Data Parameters
NAME AB374
EXPNO 10
PROCNO 1

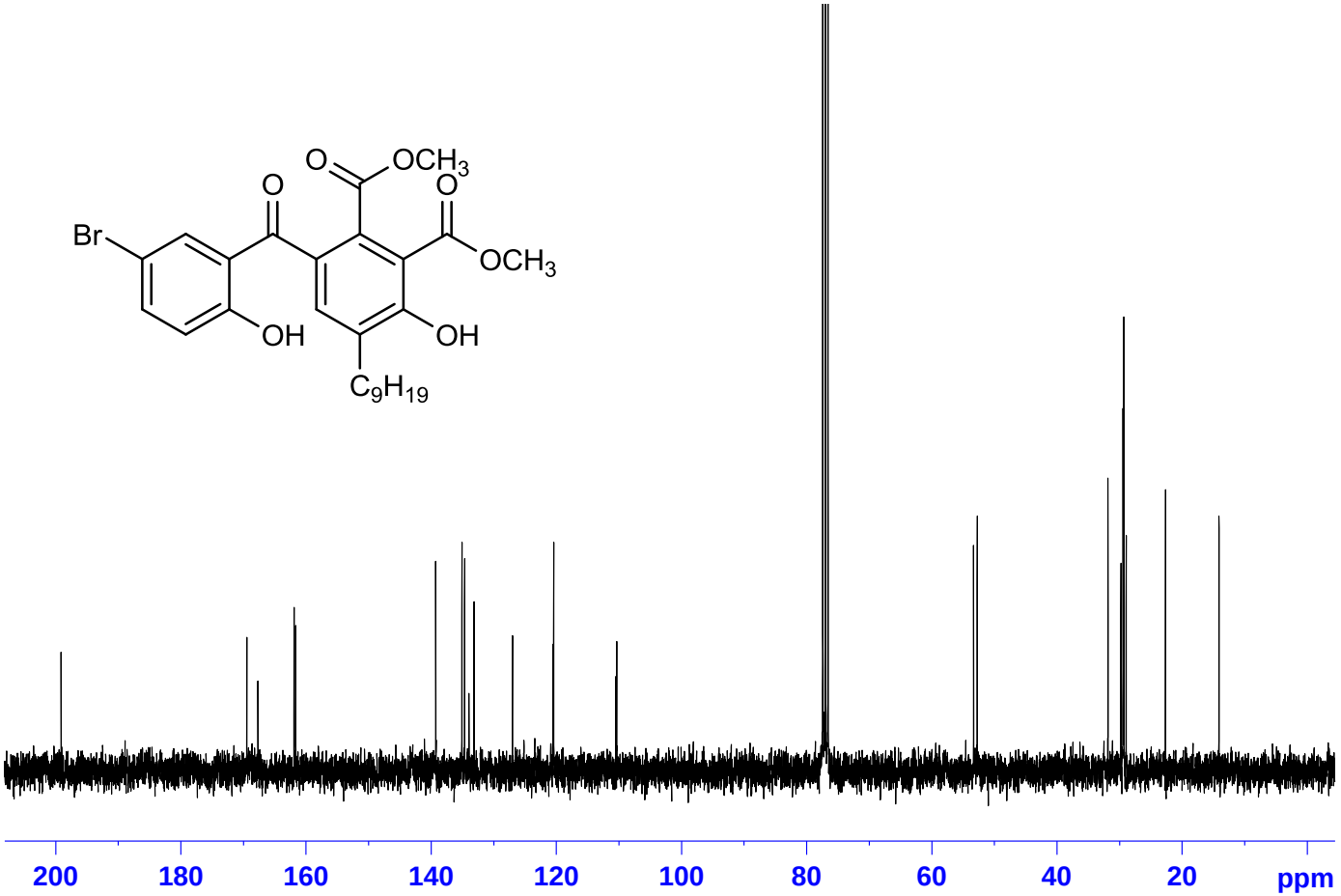
F2 - Acquisition Parameters
Date_ 20110203
Time 18.04
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 4
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 143.7
DW 81.000 usec
DE 6.00 usec
TE 294.2 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 0.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300151 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

Bunescu AB374
Au13C CDCl3 /opt/topspin 1102 7

199.16
169.47
167.71
161.93
161.66
139.28
135.10
134.66
133.97
133.16
127.02
120.49
120.43
110.51
110.37
53.34
52.75
31.85
29.78
29.45
29.31
28.94
22.65
14.10



Current Data Parameters
NAME AB374
EXPNO 11
PROCNO 1

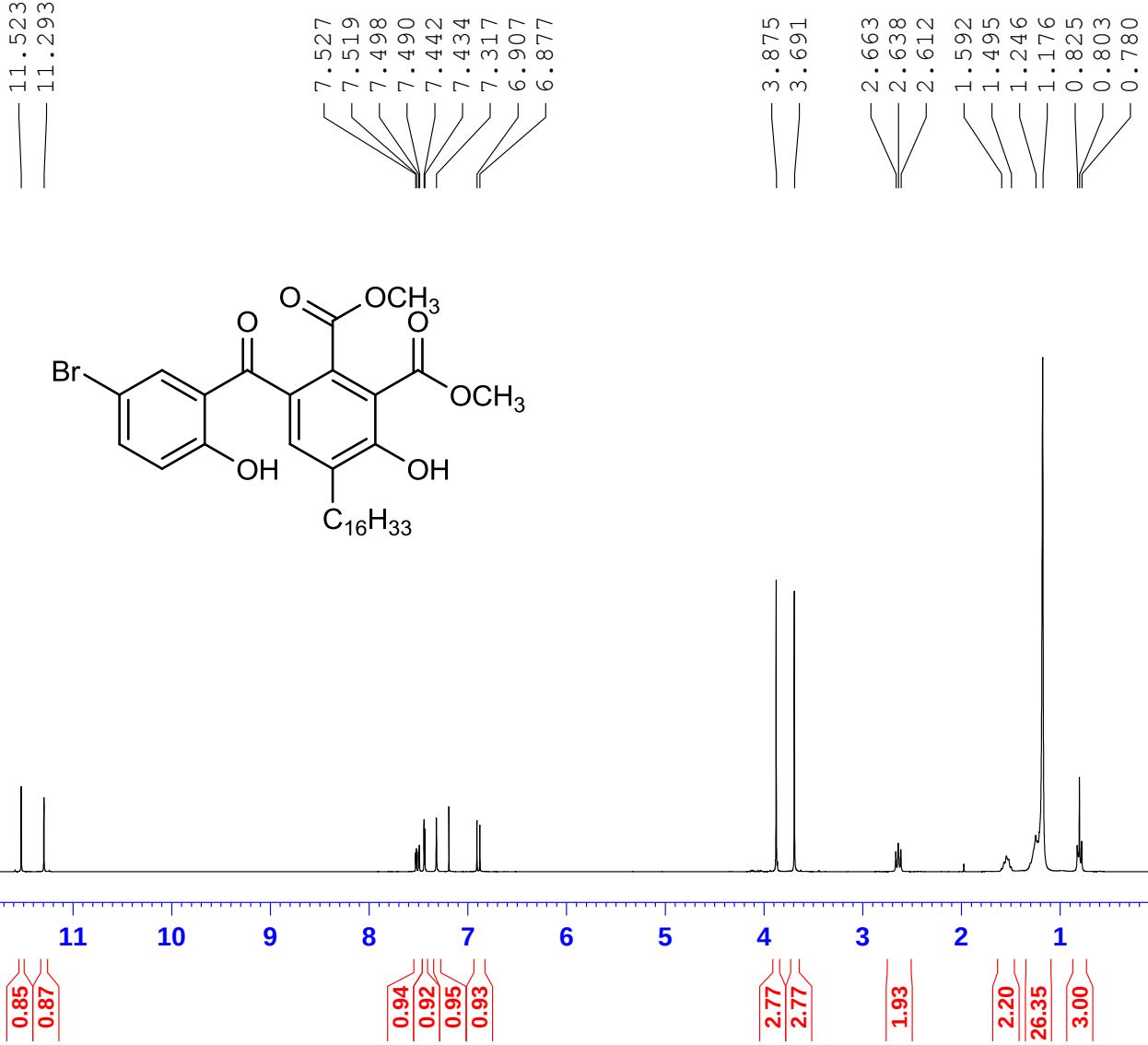
F2 - Acquisition Parameters
Date_ 20110203
Time 18.17
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 256
DS 4
SWH 21097.047 Hz
FIDRES 0.643831 Hz
AQ 0.7766516 sec
RG 32768
DW 23.700 usec
DE 6.00 usec
TE 294.7 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.60 usec
PL1 -1.10 dB
SFO1 75.4771825 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 19.00 dB
PL13 21.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677532 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Bunescu AB372
Au1H CDCl3 /opt/topspin 1102 54



Current Data Parameters
NAME AB372
EXPNO 10
PROCNO 1

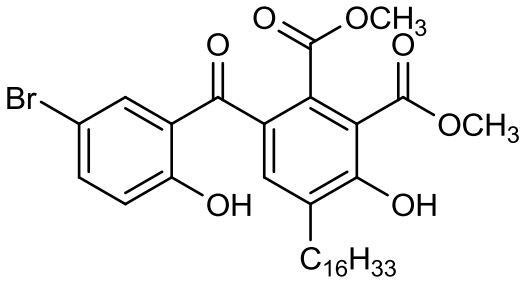
F2 - Acquisition Parameters
Date_ 20110212
Time 18.06
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 4
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 128
DW 81.000 usec
DE 6.00 usec
TE 294.3 K
D1 1.00000000 sec
TD0 1

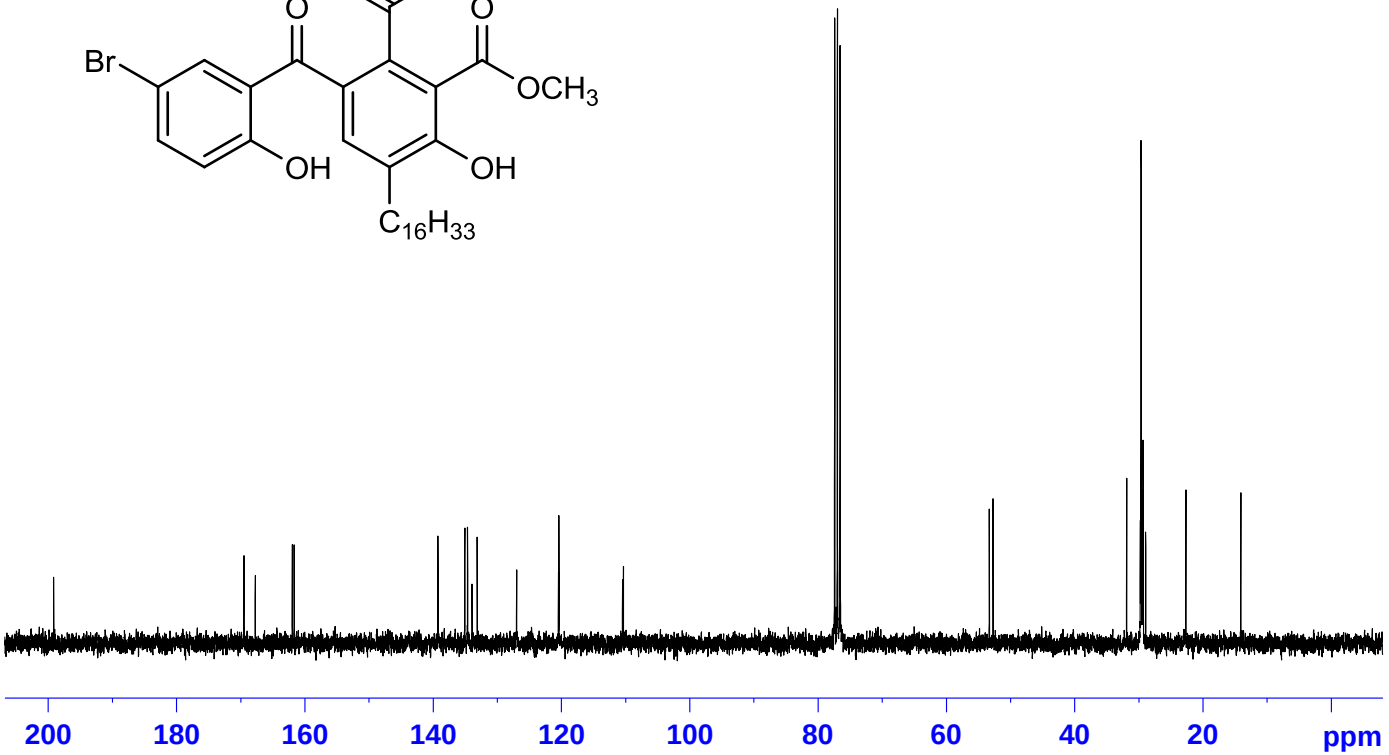
======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 0.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300362 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

Bunescu AB372
Au13C CDCl3 /opt/topspin 1102 54

199.15
169.46
167.71
161.93
161.66
139.28
135.09
134.66
133.97
133.15
127.01
120.49
120.43
110.50
110.36
53.33
52.74
31.91
29.78
29.68
29.65
29.51
29.45
29.35
28.94
22.68
14.12





Current Data Parameters
NAME AB372
EXPNO 11
PROCNO 1

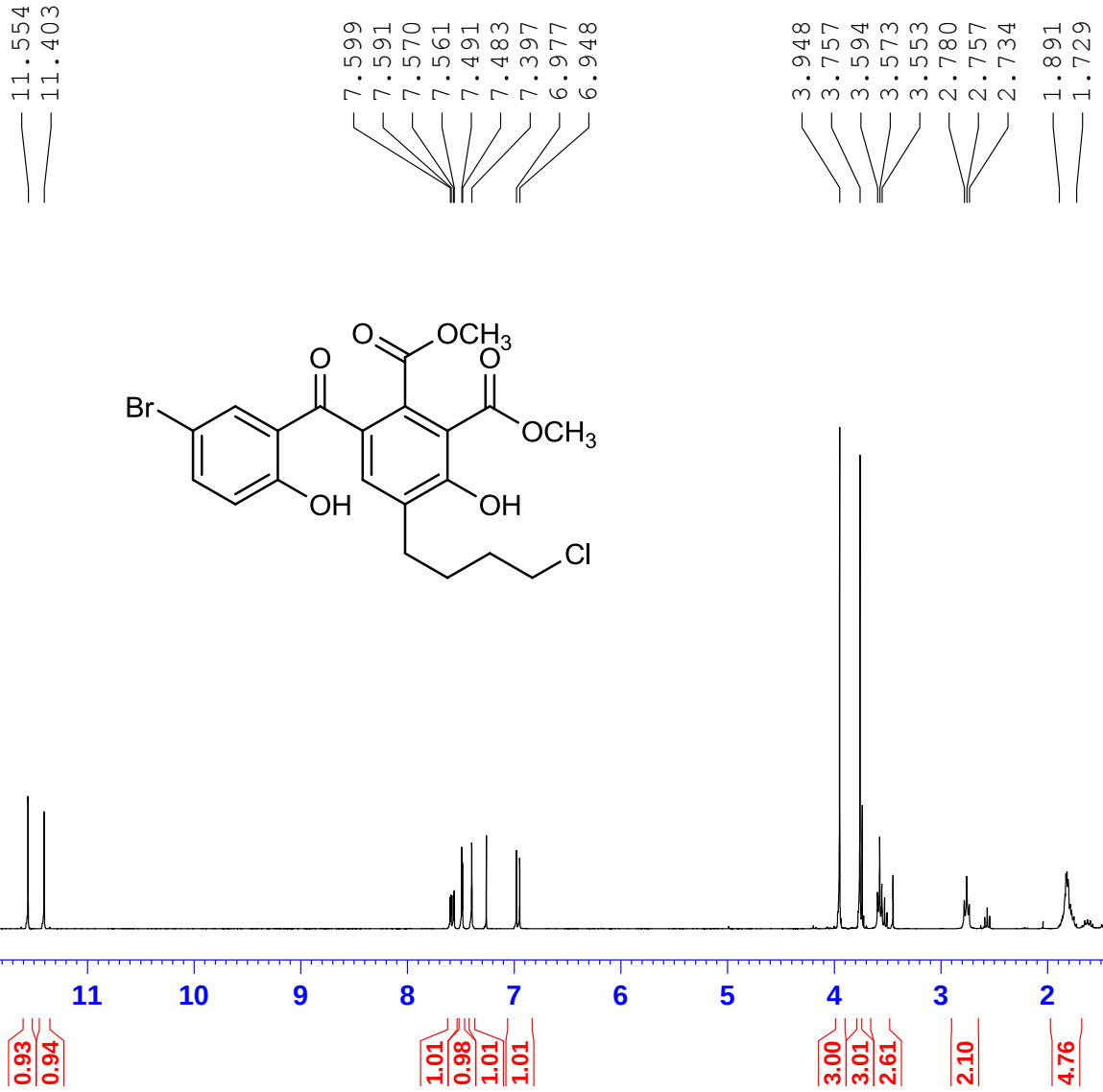
F2 - Acquisition Parameters
Date_ 20110212
Time 18.20
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 256
DS 4
SWH 21097.047 Hz
FIDRES 0.643831 Hz
AQ 0.7766516 sec
RG 32768
DW 23.700 usec
DE 6.00 usec
TE 294.9 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.60 usec
PL1 -1.10 dB
SFO1 75.4771825 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 19.00 dB
PL13 21.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677536 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Bunescu AB375
Au1H CDCl3 /opt/topspin 1102 46



Current Data Parameters
NAME AB375 1H
EXPNO 10
PROCNO 1

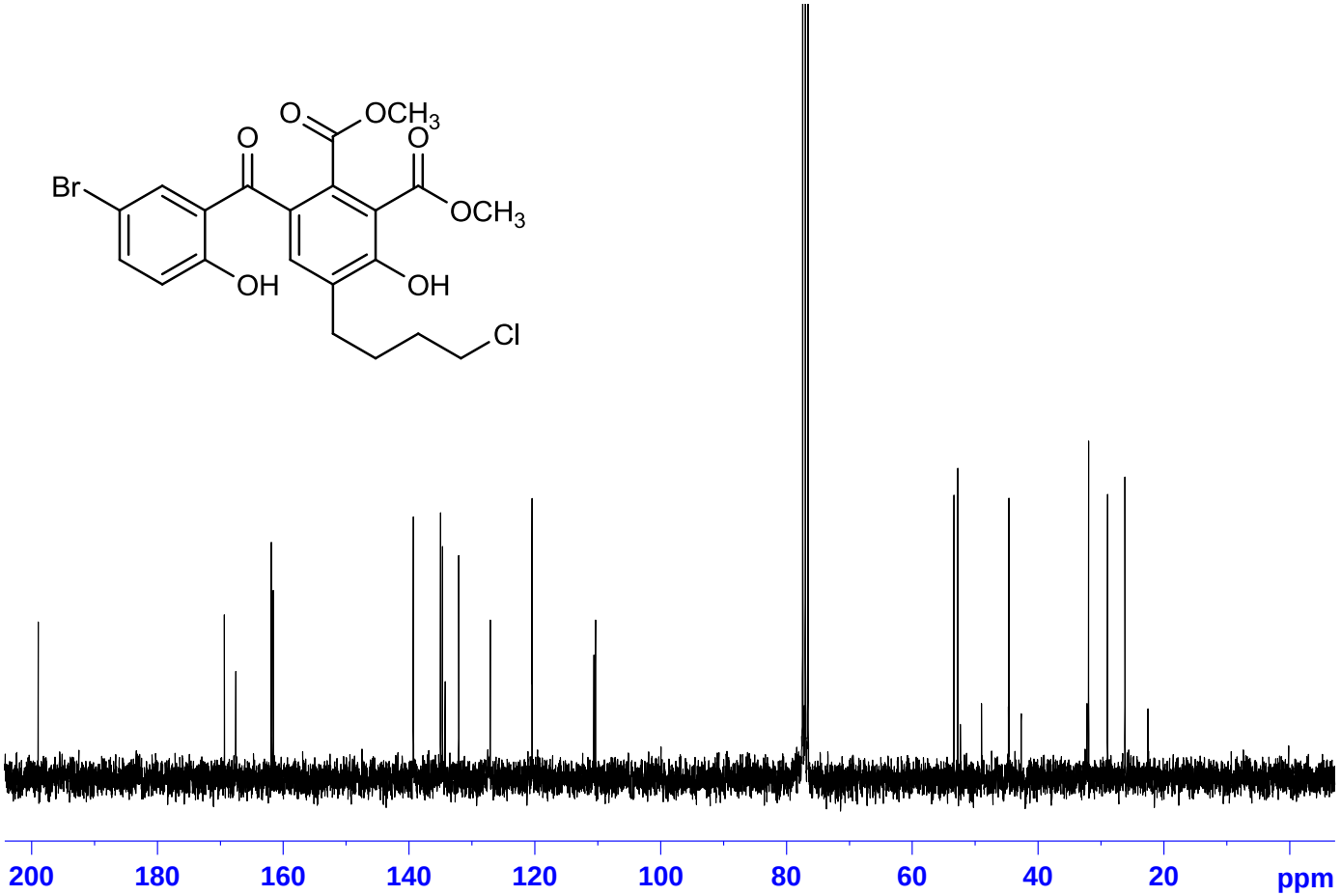
F2 - Acquisition Parameters
Date_ 20110224
Time 0.52
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 4
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 181
DW 81.000 usec
DE 6.00 usec
TE 294.2 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 0.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300150 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

Bunescu AB375
Au13C CDCl3 /opt/topspin 1102 46

198.97
169.37
167.55
161.91
161.61
139.33
135.01
134.70
134.28
132.12
127.10
120.46
120.43
110.63
110.37
53.40
52.77
44.66
31.98
28.97
26.20



Current Data Parameters
NAME AB375 13C
EXPNO 11
PROCNO 1

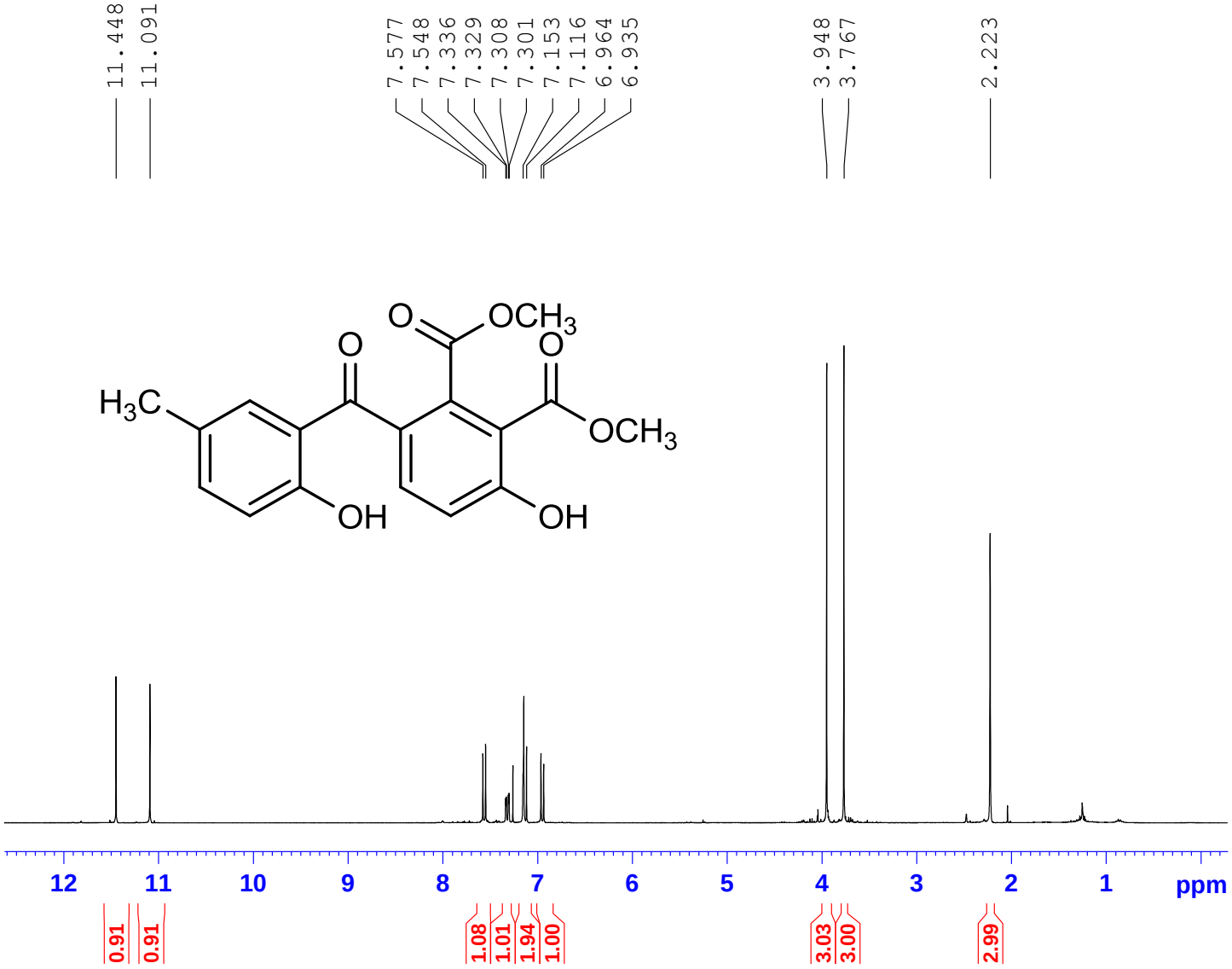
F2 - Acquisition Parameters
Date_ 20110224
Time 1.06
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 256
DS 4
SWH 21097.047 Hz
FIDRES 0.643831 Hz
AQ 0.7766516 sec
RG 32768
DW 23.700 usec
DE 6.00 usec
TE 294.7 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.60 usec
PL1 -1.10 dB
SFO1 75.4771825 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 19.00 dB
PL13 21.00 dB
SFO2 300.1312005 MHz

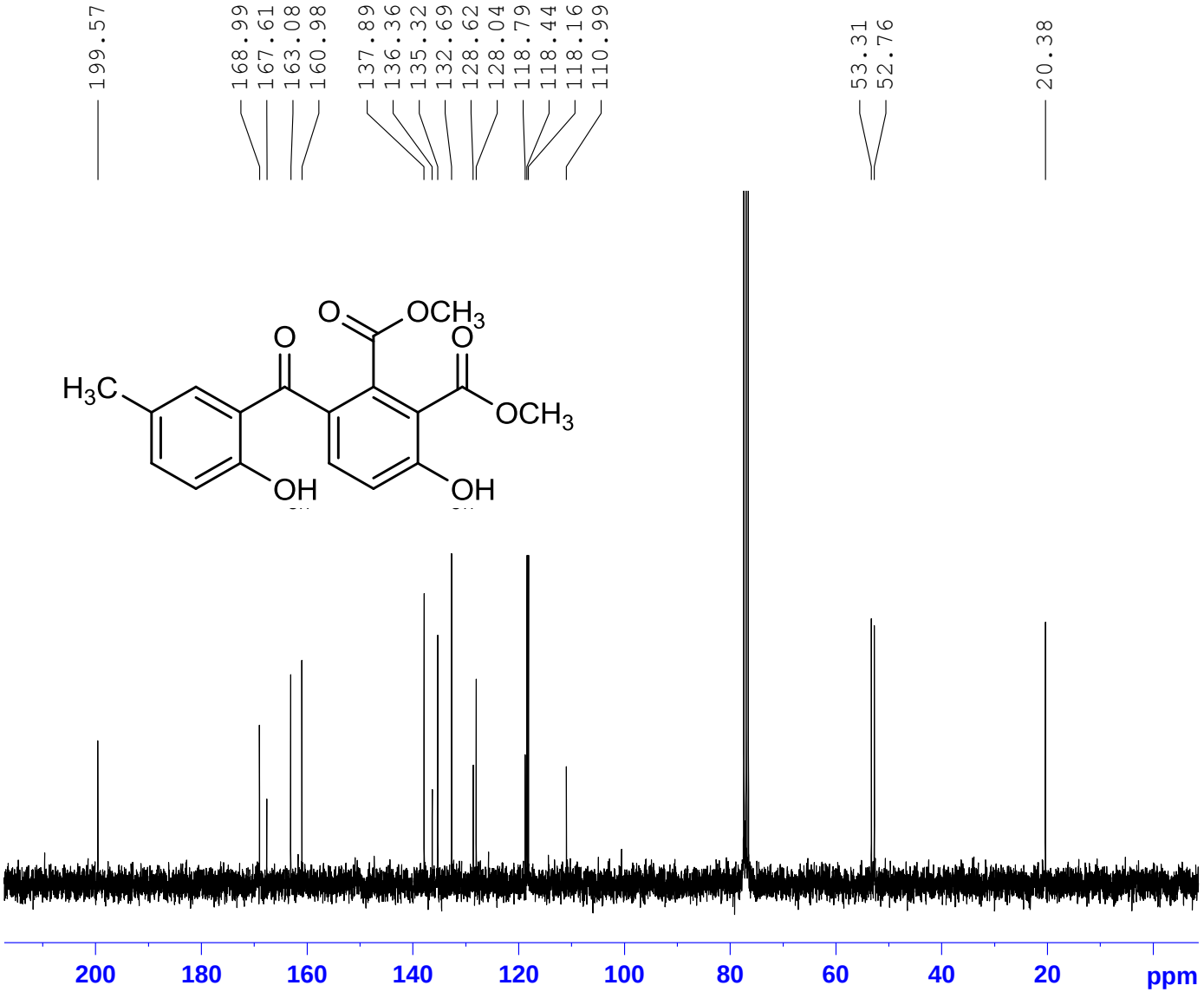
F2 - Processing parameters
SI 32768
SF 75.4677547 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Bunescu AB367B
Au1H CDCl3 /opt/topspin 1101 19




```
Current Data Parameters
NAME                  AB367B
EXPNO                     10
PROCNO                     1

F2 - Acquisition Parameters
Date_              20110117
Time                  15.49
INSTRUM               AV300
PROBHD    5 mm PABBO BB-
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       32
DS                        4
SWH               6172.839 Hz
FIDRES            0.188380 Hz
AQ                2.6542580 sec
RG                      181
DW                 81.000 usec
DE                  6.00 usec
TE                  297.0 K
D1              1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    1H
P1                  11.00 usec
PL1                  0.00 dB
SFO1          300.1318534 MHz

F2 - Processing parameters
SI                    32768
SF            300.1300152 MHz
WDW                      EM
SSB                       0
LB                   0.10 Hz
GB                        0
PC                     1.00
```

Bunescu AB367B
Au13C CDCl3 /opt/topspin 1101 19



Current Data Parameters
NAME AB367 B
EXPNO 11
PROCNO 1

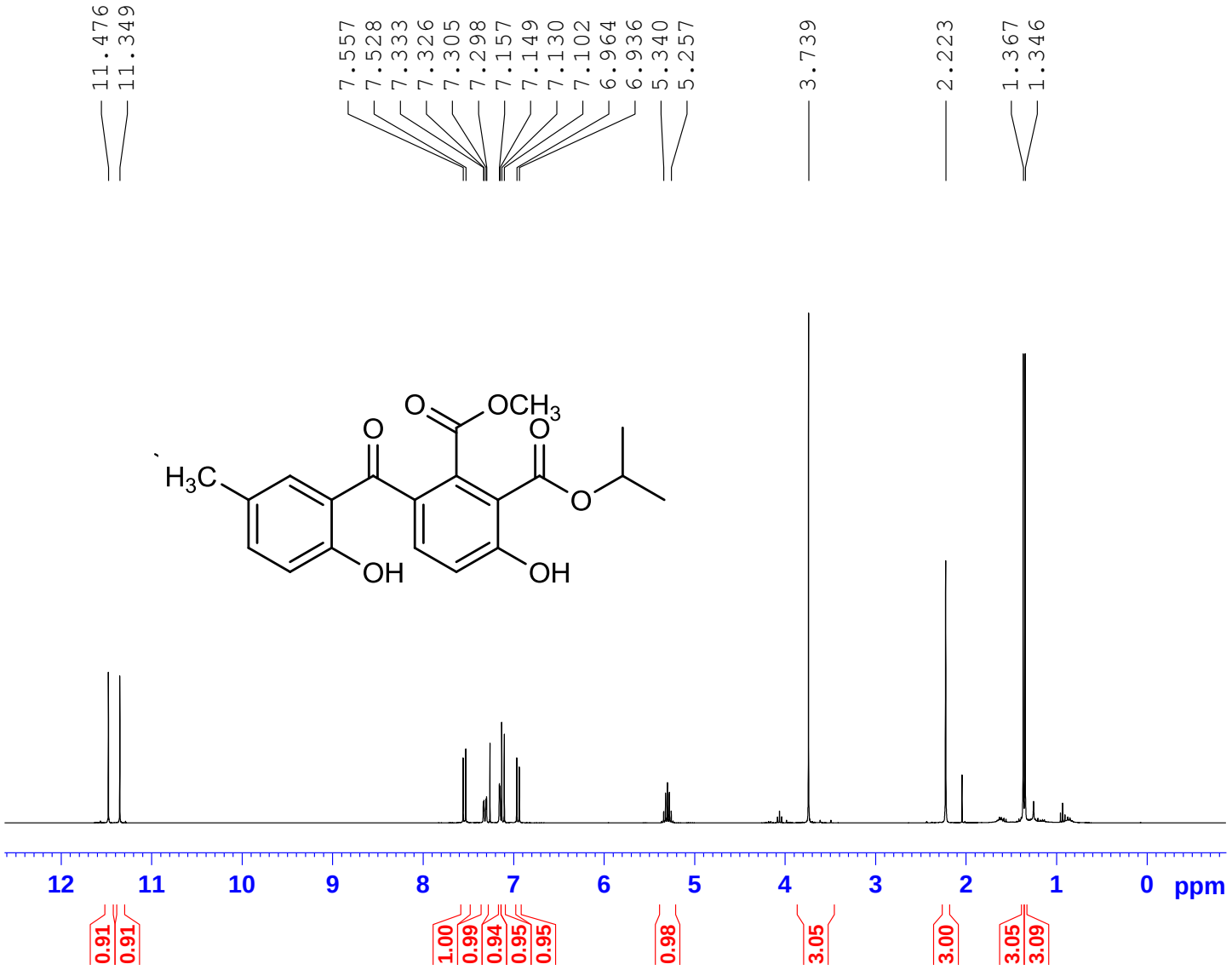
F2 - Acquisition Parameters
Date_ 20110117
Time 16.02
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 256
DS 4
SWH 21097.047 Hz
FIDRES 0.643831 Hz
AQ 0.7766516 sec
RG 32768
DW 23.700 usec
DE 6.00 usec
TE 297.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.60 usec
PL1 -1.10 dB
SFO1 75.4771825 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 19.00 dB
PL13 21.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677542 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Bunescu AB385
Au1H CDCl3 /opt/topspin 1104 35



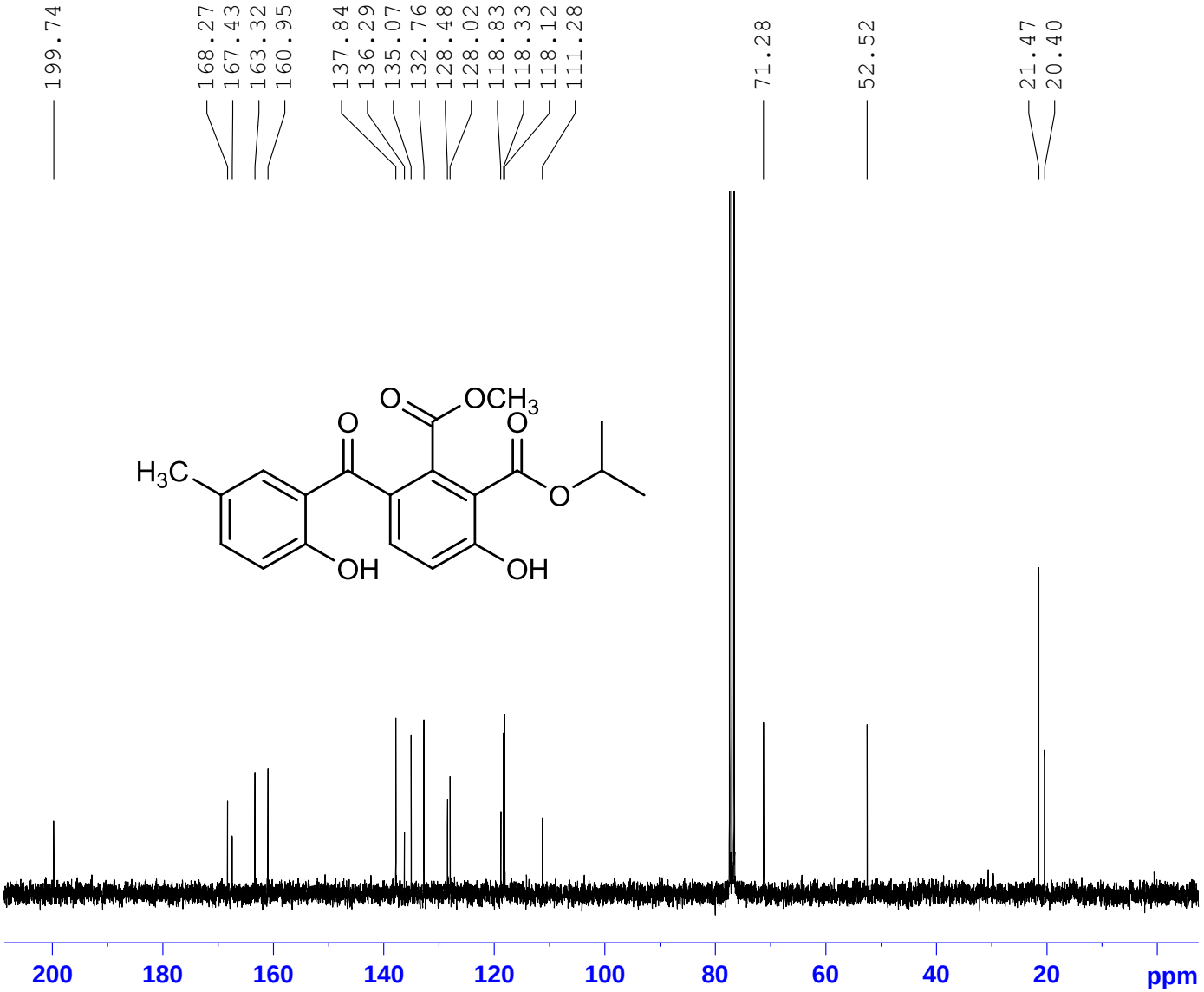
Current Data Parameters
NAME AB385B 1H
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110407
Time 3.57
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 4
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 161.3
DW 81.000 usec
DE 6.00 usec
TE 294.8 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 0.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300152 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

Bunescu AB385
Au13C CDCl3 /opt/topspin 1105 31



Current Data Parameters
NAME AB385B 13C
EXPNO 10
PROCNO 1

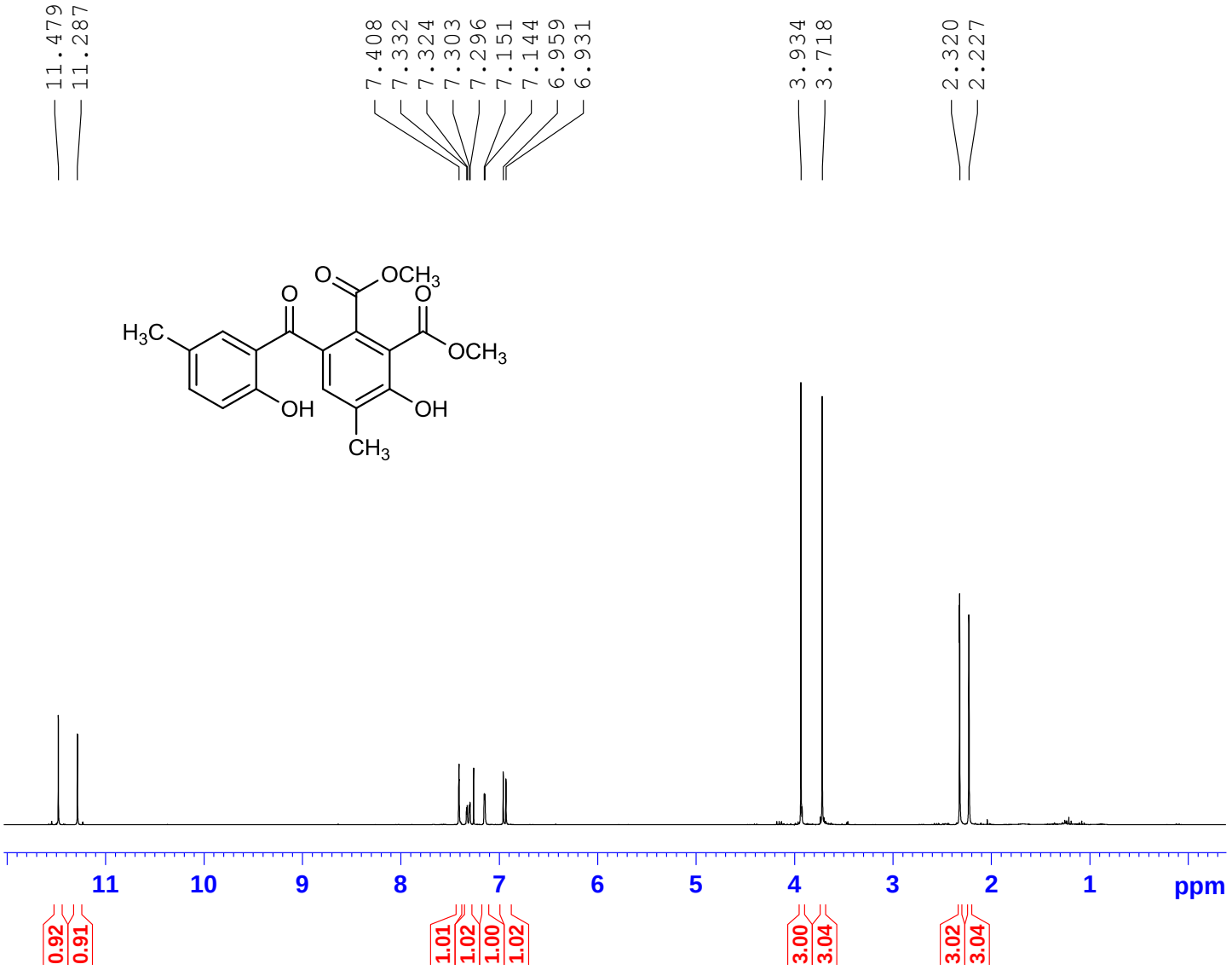
F2 - Acquisition Parameters
Date_ 20110506
Time 0.42
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 256
DS 4
SWH 21097.047 Hz
FIDRES 0.643831 Hz
AQ 0.7766516 sec
RG 32768
DW 23.700 usec
DE 6.00 usec
TE 295.9 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.60 usec
PL1 -1.10 dB
SFO1 75.4771825 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 19.00 dB
PL13 21.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677534 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Bunescu AB376
Au1H CDCl3 /opt/topspin 1102 12



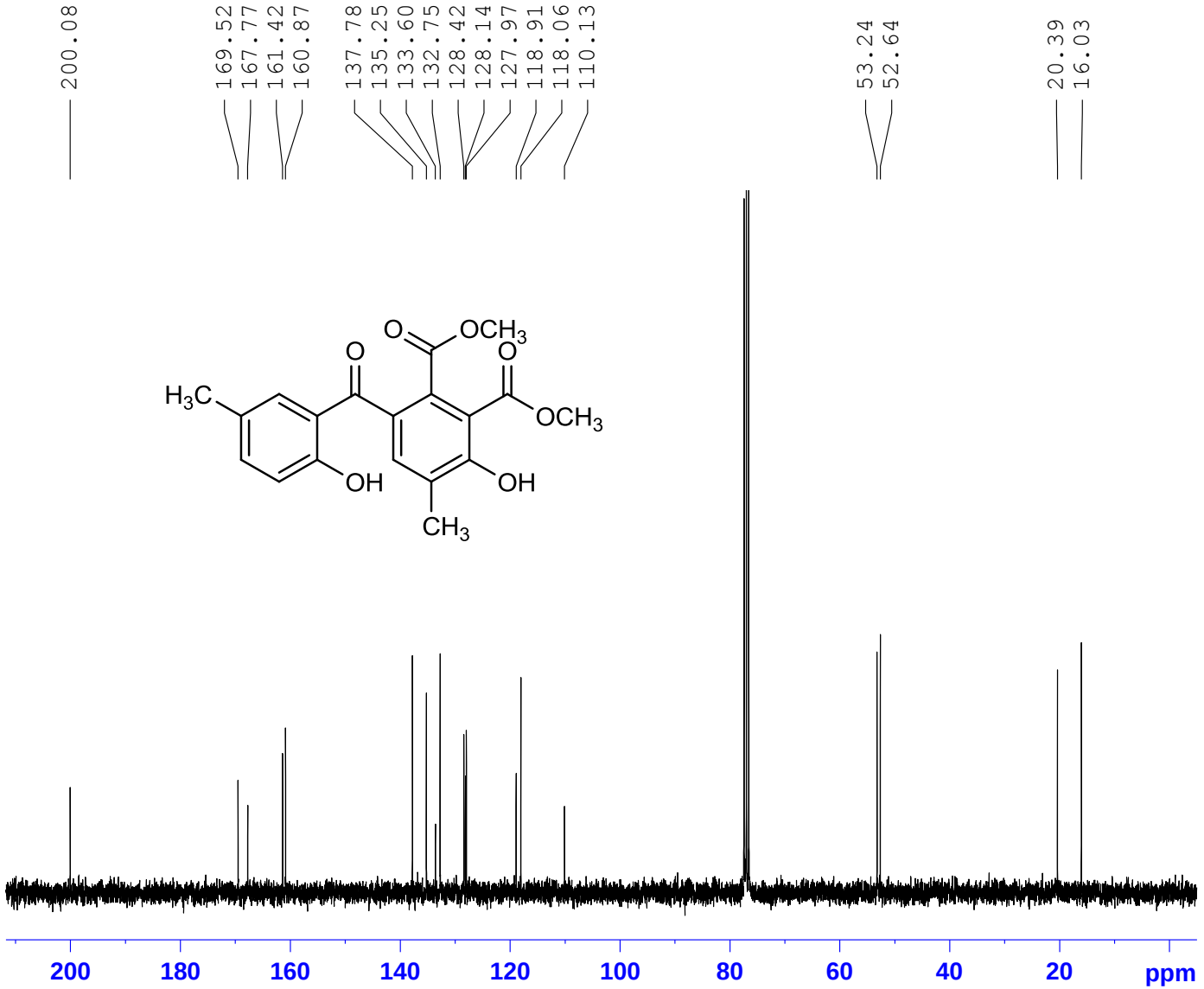
Current Data Parameters
NAME AB376
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110209
Time 21.01
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 4
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 161.3
DW 81.000 usec
DE 6.00 usec
TE 293.9 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 0.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300152 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

Bunescu AB376
Au13C CDCl3 /opt/topspin 1102 12



Current Data Parameters
NAME AB376
EXPNO 11
PROCNO 1

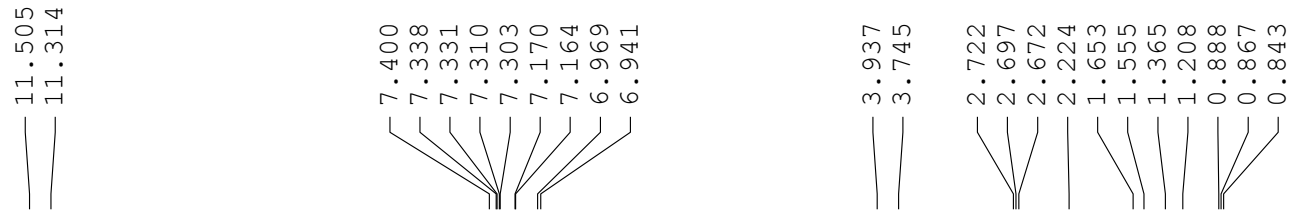
F2 - Acquisition Parameters
Date_ 20110209
Time 21.13
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 256
DS 4
SWH 21097.047 Hz
FIDRES 0.643831 Hz
AQ 0.7766516 sec
RG 32768
DW 23.700 usec
DE 6.00 usec
TE 294.3 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

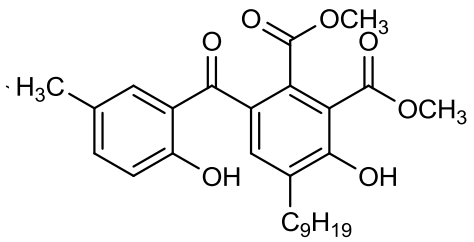
======== CHANNEL f1 ========
NUC1 13C
P1 9.60 usec
PL1 -1.10 dB
SFO1 75.4771825 MHz

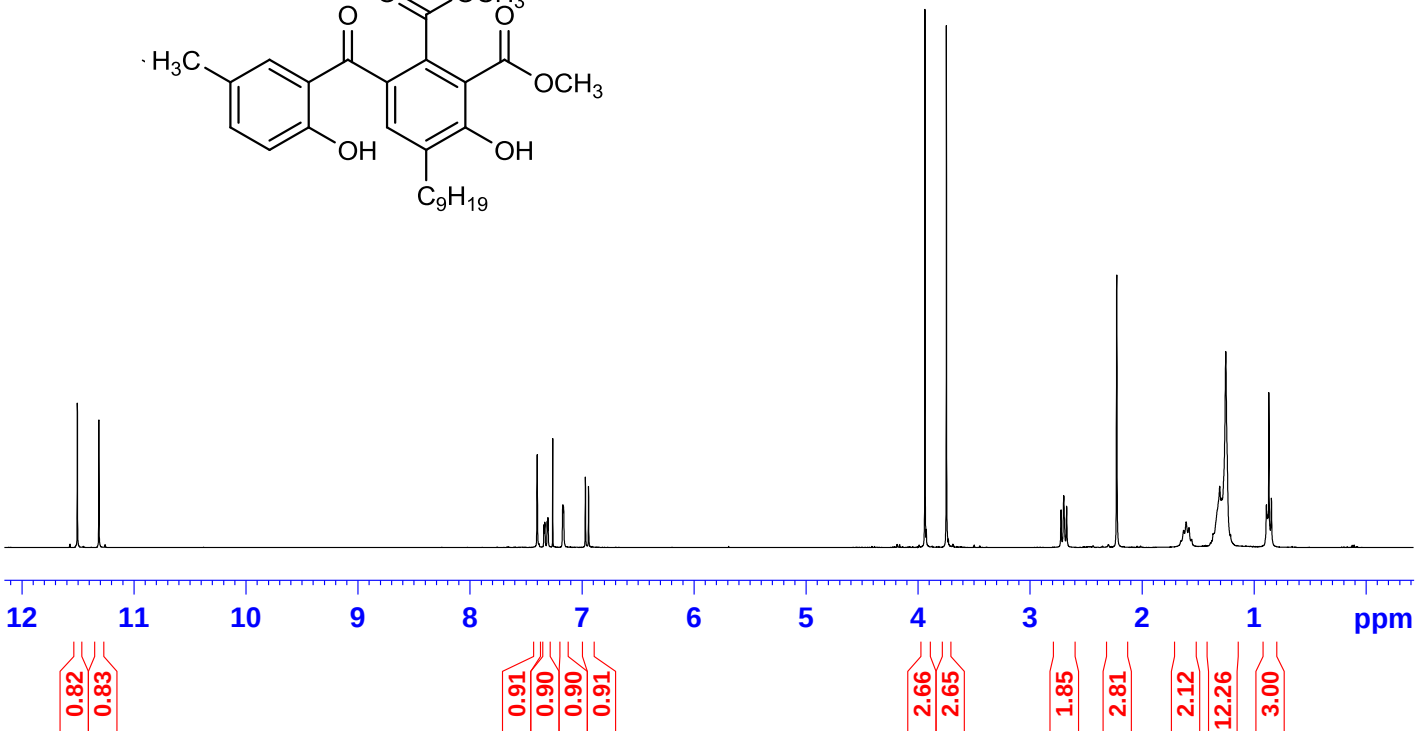
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 19.00 dB
PL13 21.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677546 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Bunescu AB378
Au1H CDCl3 /opt/topspin 1102 55







Current Data Parameters
NAME AB378
EXPNO 10
PROCNO 1

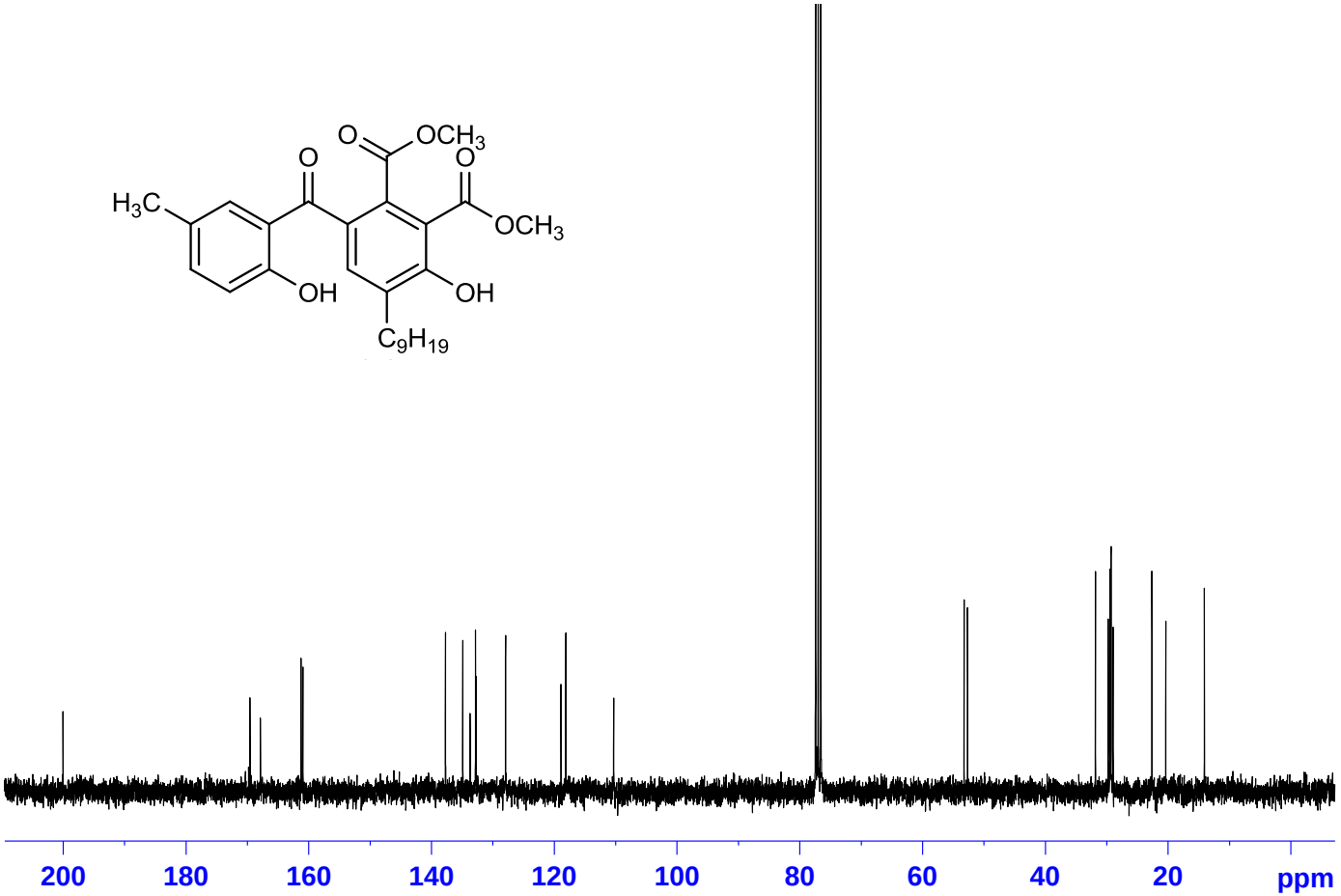
F2 - Acquisition Parameters
Date_ 20110212
Time 18.37
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 4
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 143.7
DW 81.000 usec
DE 6.00 usec
TE 294.3 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 0.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300152 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

Bunescu AB378
Au13C CDCl3 /opt/topspin 1102 55

200.05
169.59
167.90
161.28
160.97
137.74
134.92
133.72
132.84
132.72
127.95
127.94
118.91
118.12
110.34
53.24
52.68
31.83
29.76
29.48
29.44
29.31
29.29
28.96
22.63
20.37
14.08



Current Data Parameters
NAME AB378
EXPNO 11
PROCNO 1

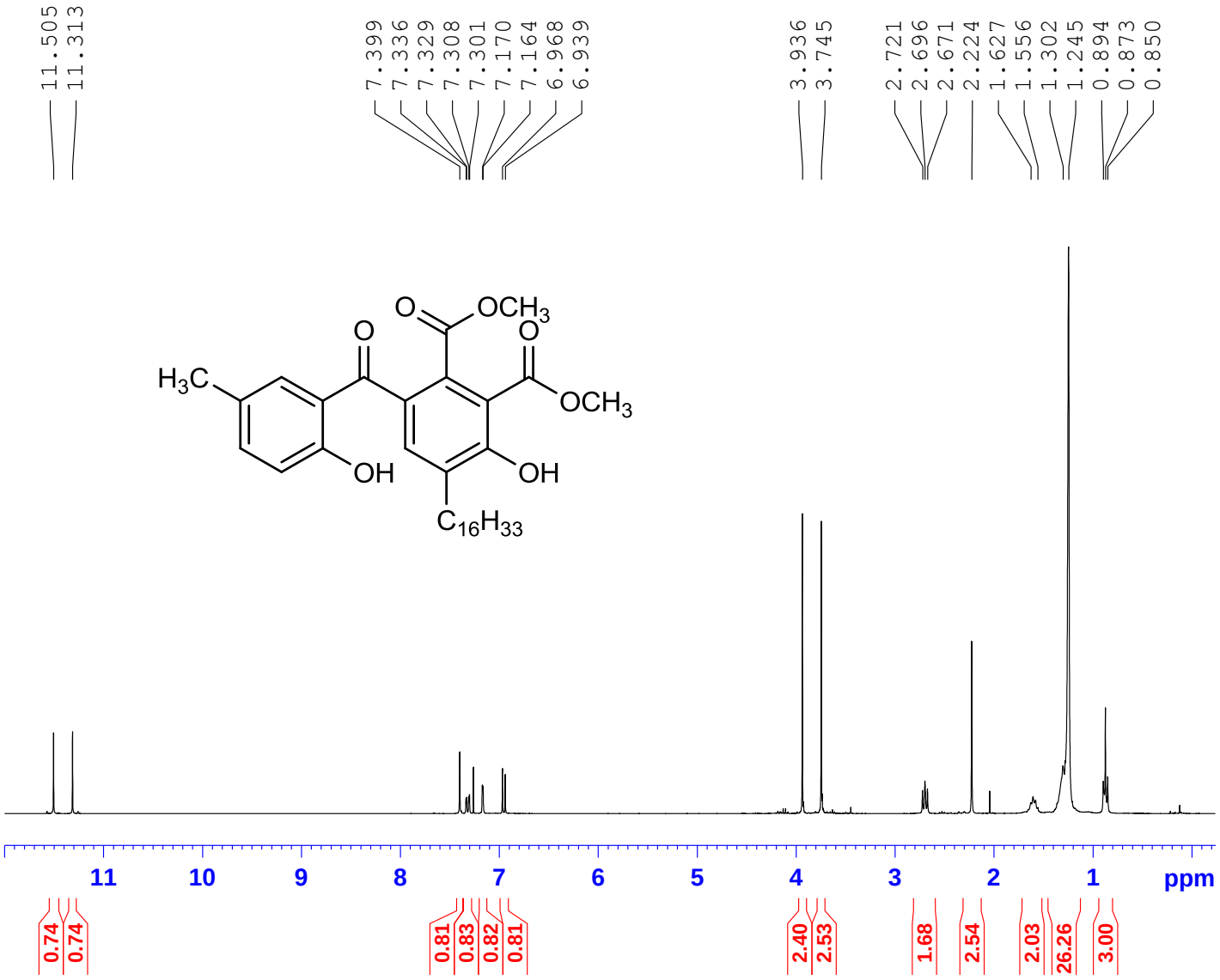
F2 - Acquisition Parameters
Date_ 20110212
Time 18.49
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 256
DS 4
SWH 21097.047 Hz
FIDRES 0.643831 Hz
AQ 0.7766516 sec
RG 32768
DW 23.700 usec
DE 6.00 usec
TE 294.8 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.60 usec
PL1 -1.10 dB
SFO1 75.4771825 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 19.00 dB
PL13 21.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677538 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

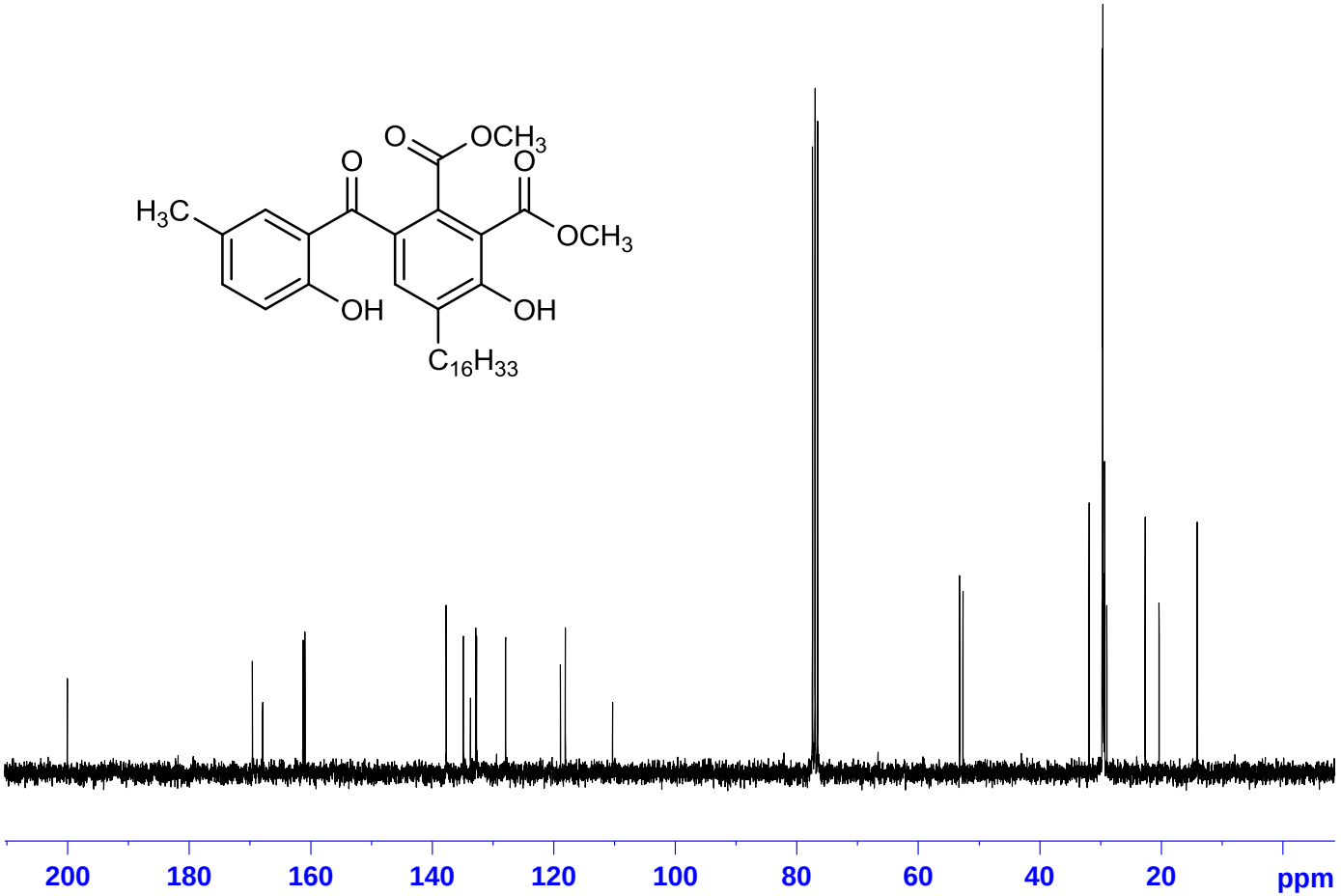
Bunescu AB377
Au1H CDCl3 /opt/topspin 1102 52




```
Current Data Parameters
NAME                  AB377
EXPNO                    10
PROCNO                    1

F2 - Acquisition Parameters
Date_              20110206
Time                 16.48
INSTRUM              AV300
PROBHD    5 mm PABBO BB-
PULPROG               zg30
TD                   32768
SOLVENT              CDCl3
NS                      32
DS                       4
SWH               6172.839 Hz
FIDRES            0.188380 Hz
AQ                2.6542580 sec
RG                    90.5
DW                  81.000 usec
DE                   6.00 usec
TE                  294.3 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   11.00 usec
PL1                   0.00 dB
SFO1          300.1318534 MHz

F2 - Processing parameters
SI                   32768
SF            300.1300152 MHz
WDW                     EM
SSB                      0
LB                    0.10 Hz
GB                       0
PC                    1.00
```

Bunescu AB377
Au13C CDCl3 /opt/topspin 1102 52

200.04
169.59
167.89
161.28
160.97
137.73
134.91
133.72
132.84
132.72
127.95
127.93
118.91
118.12
110.33
53.23
52.67
31.90
29.76
29.67
29.64
29.54
29.46
29.34
28.97
22.67
20.37
14.11



Current Data Parameters
NAME AB377
EXPNO 11
PROCNO 1

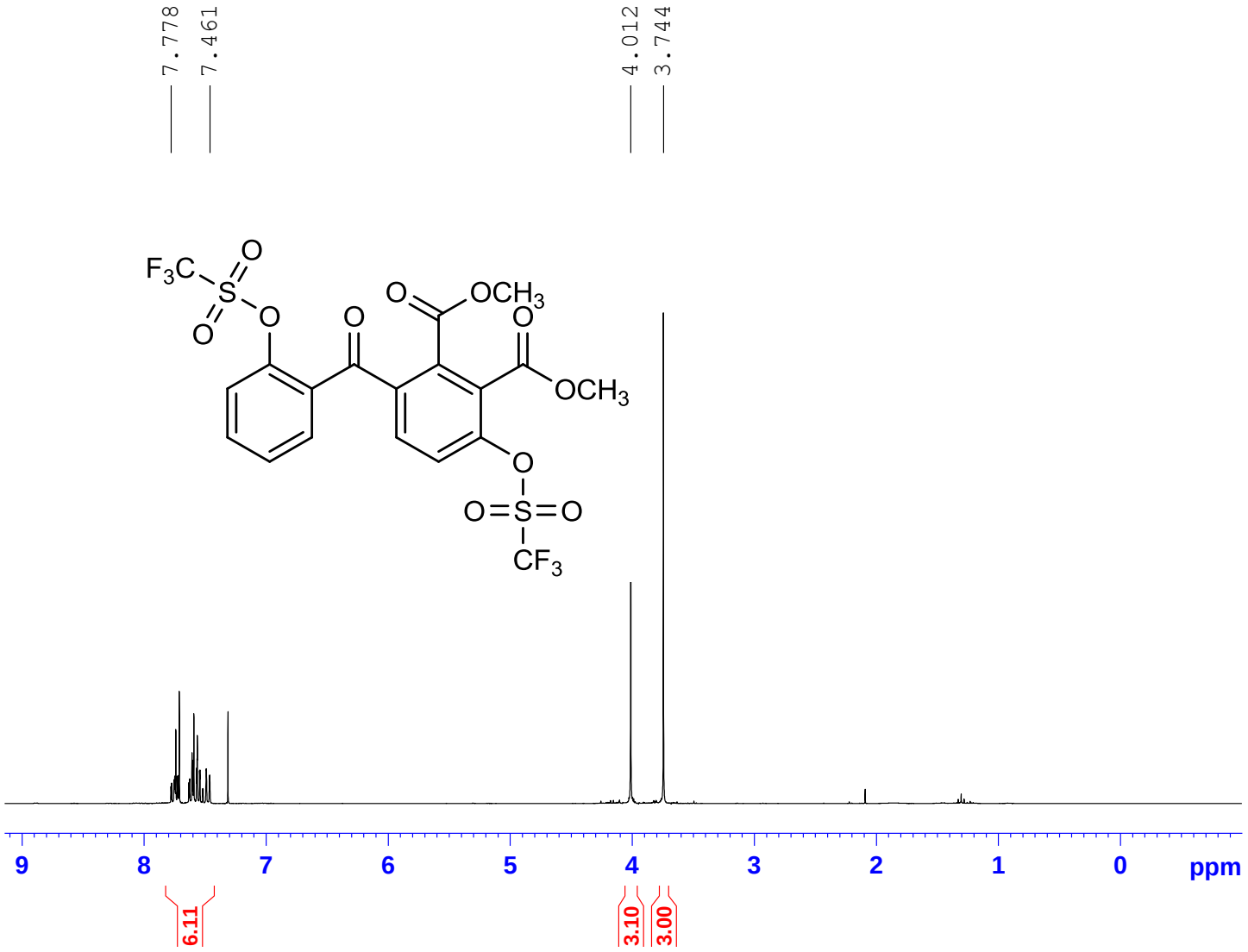
F2 - Acquisition Parameters
Date_ 20110206
Time 17.00
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 256
DS 4
SWH 21097.047 Hz
FIDRES 0.643831 Hz
AQ 0.7766516 sec
RG 32768
DW 23.700 usec
DE 6.00 usec
TE 294.8 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.60 usec
PL1 -1.10 dB
SFO1 75.4771825 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 19.00 dB
PL13 21.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677539 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

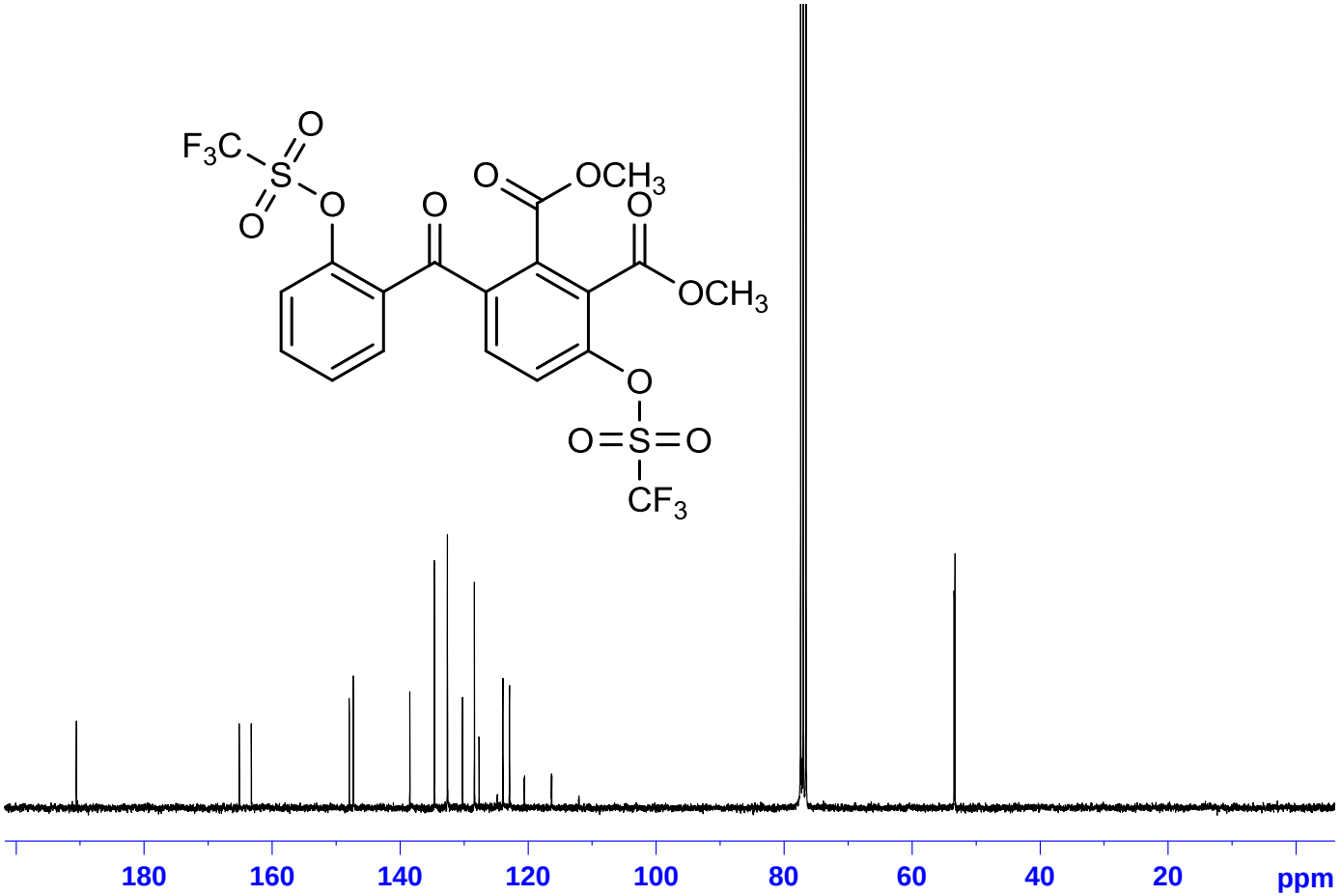
Bunescu AB347
Au1H CDCl3 /opt/topspin 1102 45




```
Current Data Parameters
NAME            AB347 1H
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20110224
Time                0.40
INSTRUM           AV300
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     4
SWH             6172.839 Hz
FIDRES          0.188380 Hz
AQ              2.6542580 sec
RG                 322.5
DW                81.000 usec
DE                 6.00 usec
TE                294.2 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                11.00 usec
PL1                0.00 dB
SFO1         300.1318534 MHz

F2 - Processing parameters
SI                 32768
SF           300.1300000 MHz
WDW                   EM
SSB                    0
LB                 0.10 Hz
GB                     0
PC                  1.00
```

Bunescu AB347
Au13C CDCl3 /opt/topspin 1102 37

190.56
165.13
163.23
147.93
147.31
138.50
134.66
132.62
130.27
128.41
127.68
124.87
124.81
123.96
122.93
120.62
120.57
116.37
116.32
112.07
53.45
53.31



Current Data Parameters
NAME AB347 13C
EXPNO 10
PROCNO 1

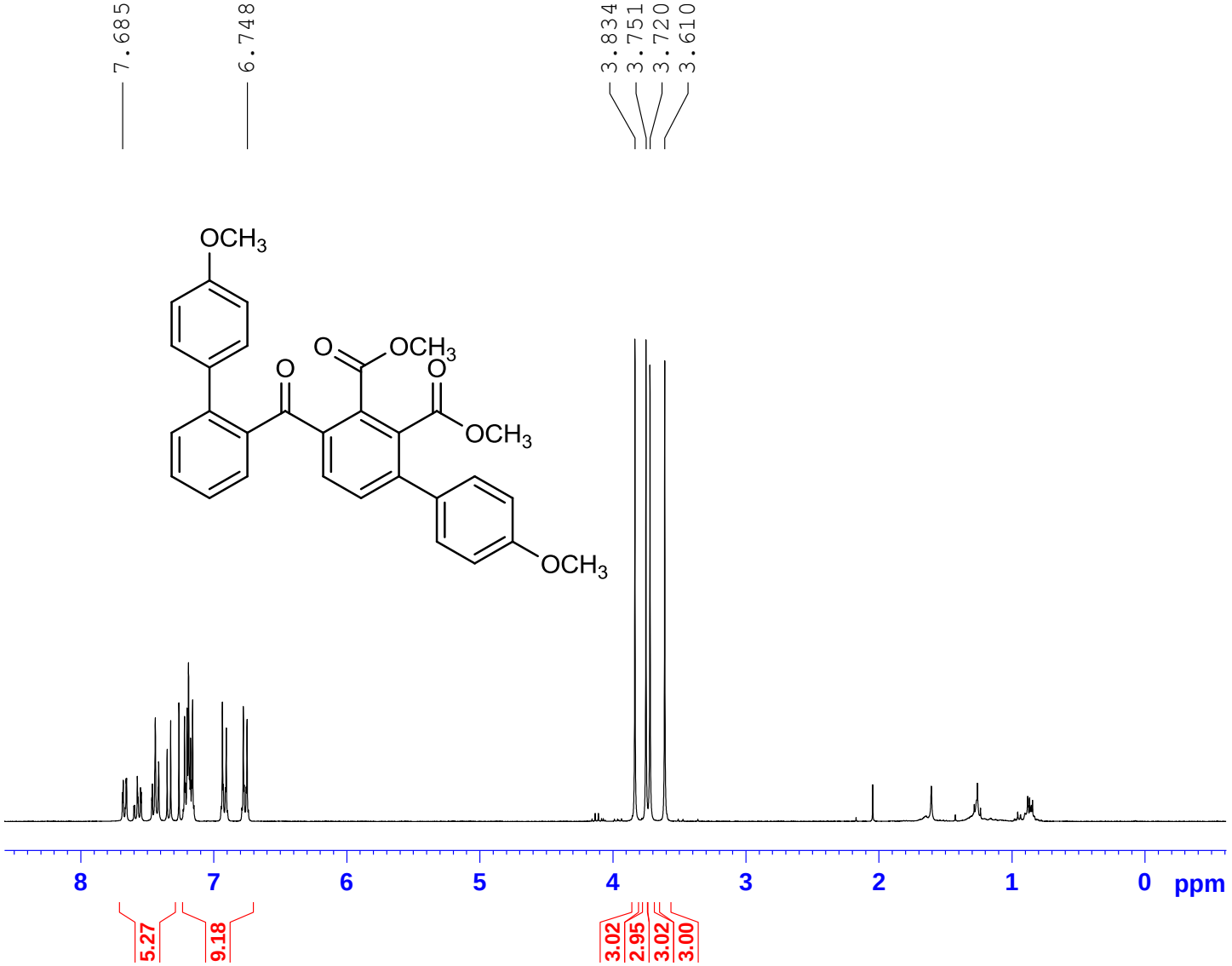
F2 - Acquisition Parameters
Date_ 20110227
Time 22.34
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 4000
DS 4
SWH 21097.047 Hz
FIDRES 0.643831 Hz
AQ 0.7766516 sec
RG 32768
DW 23.700 usec
DE 6.00 usec
TE 295.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.60 usec
PL1 -1.10 dB
SFO1 75.4771825 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 19.00 dB
PL13 21.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677531 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Bunascu AB 349
Au1H CDCl3 /opt/topspin 1011 15



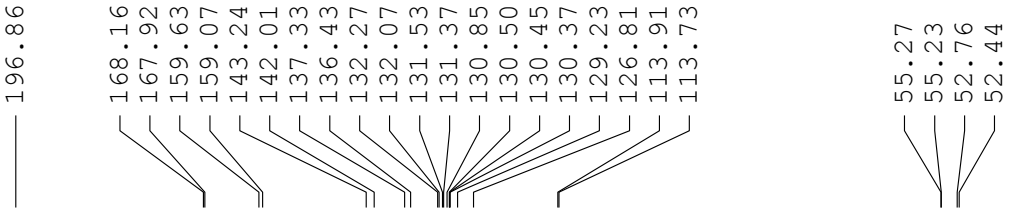
Current Data Parameters
NAME AB349
EXPNO 10
PROCNO 1

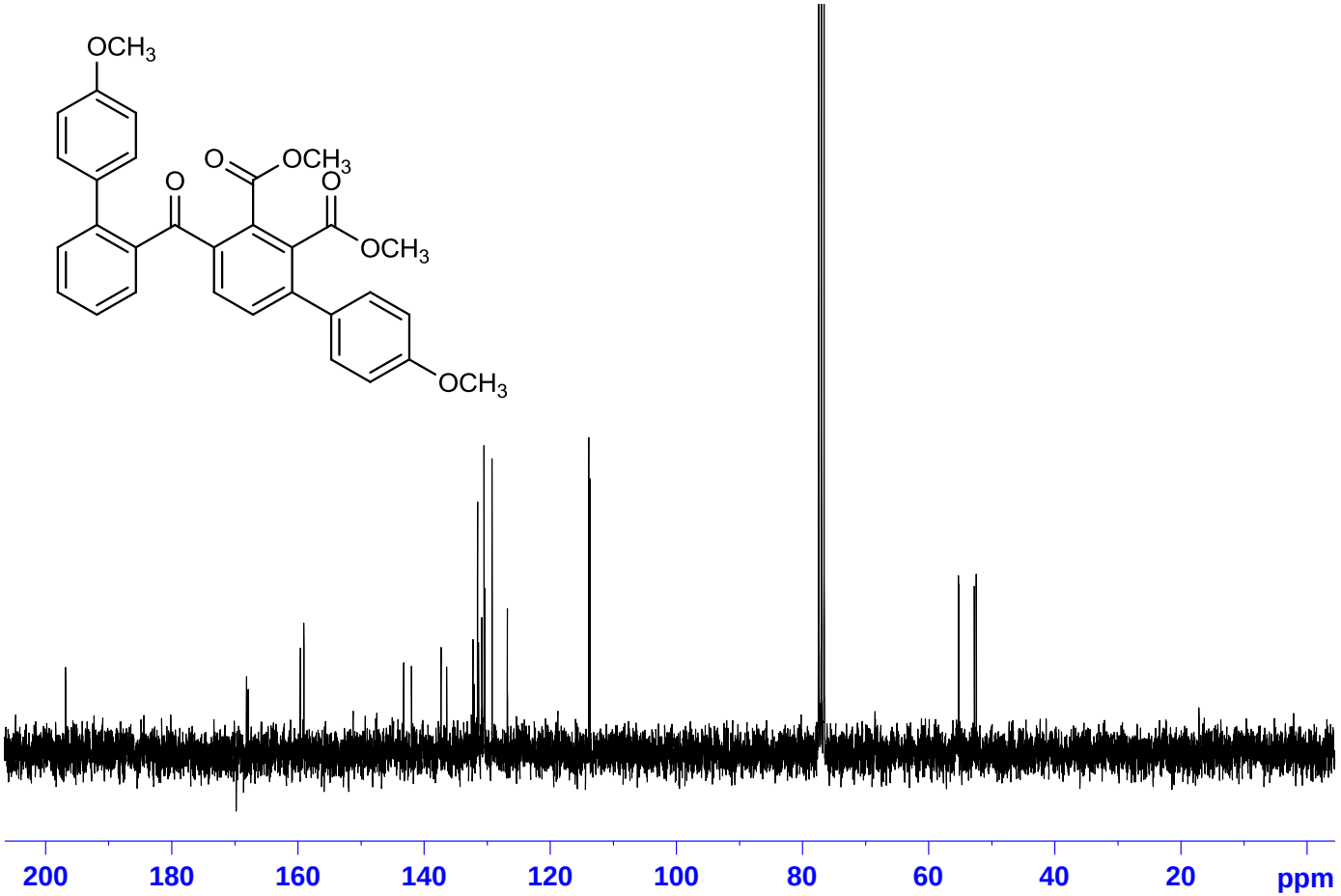
F2 - Acquisition Parameters
Date_ 20101118
Time 0.08
INSTRUM AV300
PROBHD 5 mm DUI 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 4
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 322.5
DW 81.000 usec
DE 6.00 usec
TE 293.9 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 9.70 usec
PL1 1.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300149 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

Bunascu AB 349
Au13C CDCl3 /opt/topspin 1011 15





Current Data Parameters
NAME AB349
EXPNO 11
PROCNO 1

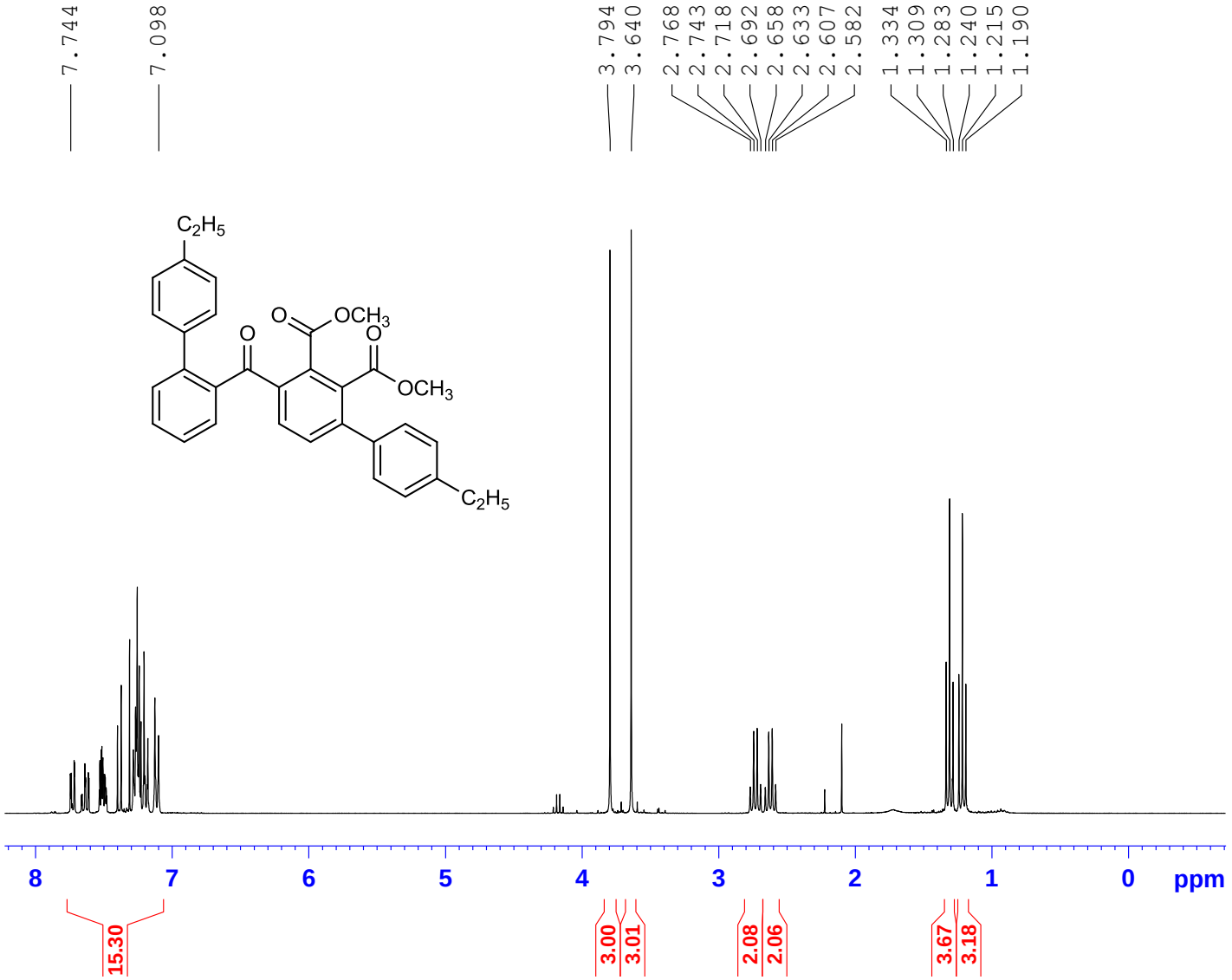
F2 - Acquisition Parameters
Date_ 20101118
Time 0.22
INSTRUM AV300
PROBHD 5 mm DUI 13C-1
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 256
DS 4
SWH 21097.047 Hz
FIDRES 0.643831 Hz
AQ 0.7766516 sec
RG 32768
DW 23.700 usec
DE 6.00 usec
TE 294.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 11.50 usec
PL1 -4.00 dB
SFO1 75.4771825 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.00 dB
PL12 21.26 dB
PL13 23.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677541 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Bunescu Ab 361
Au1H CDCl3 /opt/topspin 1012 19



Current Data Parameters
NAME AB361
EXPNO 10
PROCNO 1

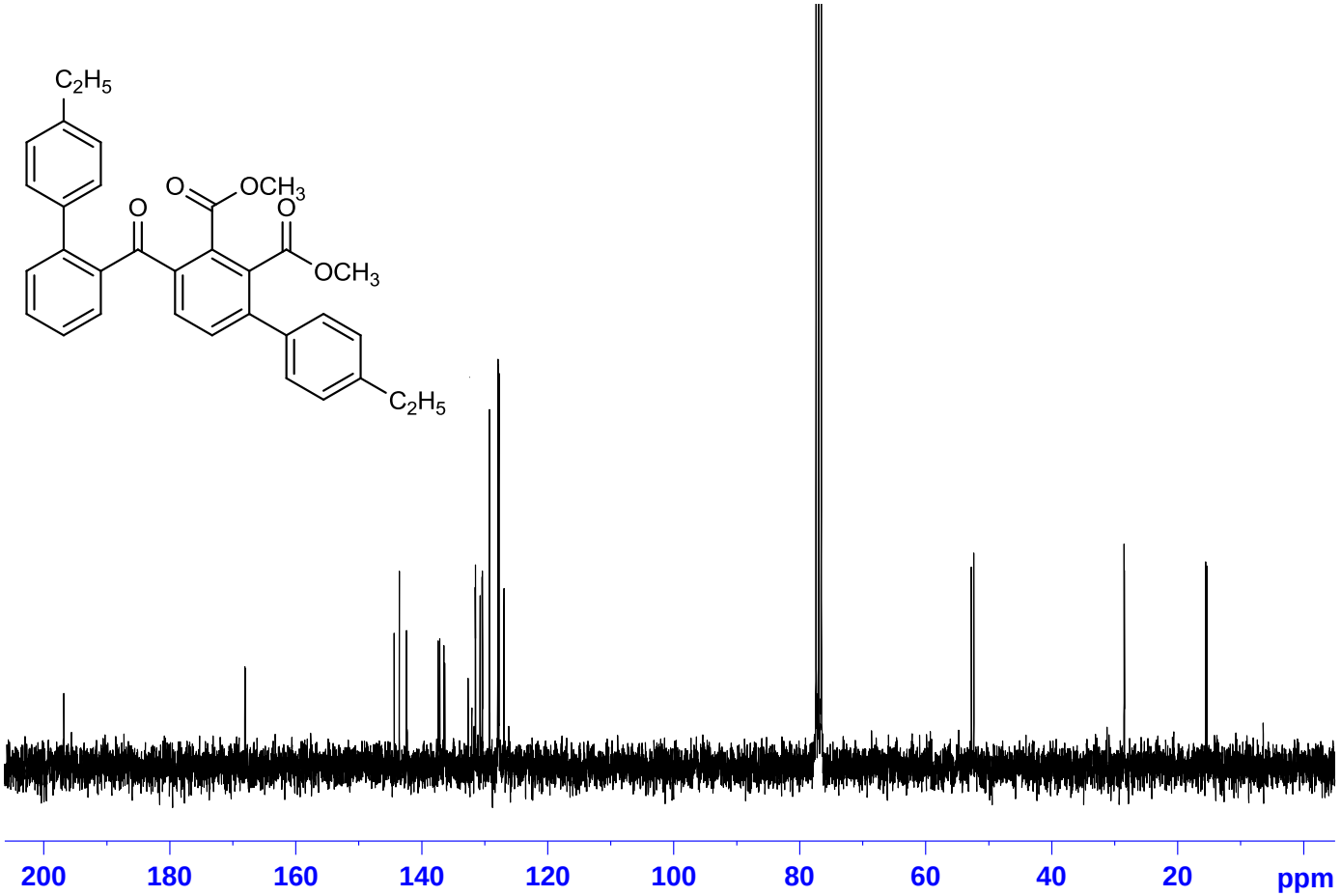
F2 - Acquisition Parameters
Date_ 20101208
Time 20.25
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 4
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 322.5
DW 81.000 usec
DE 6.00 usec
TE 294.9 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 0.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

Bunescu Ab 361
Au13C CDCl3 /opt/topspin 1012 19

196.81
168.07
167.98
144.36
143.58
142.44
137.42
137.15
136.51
136.36
132.66
132.08
131.51
131.49
130.76
130.44
130.39
129.28
127.95
127.90
127.76
126.97
52.77
52.38
28.51
28.46
15.56
15.34



Current Data Parameters
NAME AB361
EXPNO 11
PROCNO 1

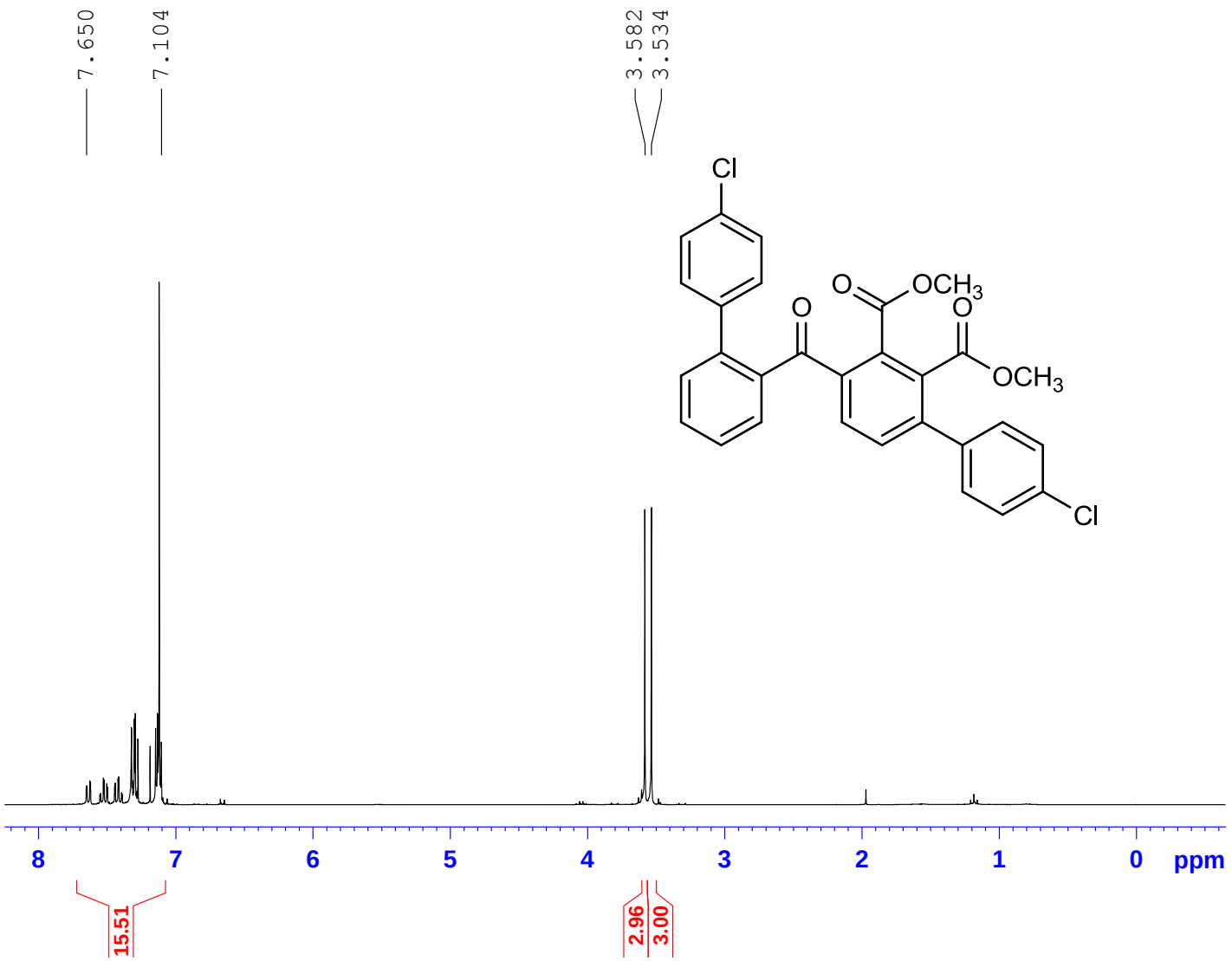
F2 - Acquisition Parameters
Date_ 20101208
Time 20.38
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 256
DS 4
SWH 21097.047 Hz
FIDRES 0.643831 Hz
AQ 0.7766516 sec
RG 32768
DW 23.700 usec
DE 6.00 usec
TE 295.4 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.60 usec
PL1 -1.10 dB
SFO1 75.4771825 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 19.00 dB
PL13 21.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677540 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Bunescu AB 365
Au1H CDCl3 /opt/topspin 1101 16



Current Data Parameters
NAME AB365
EXPNO 10
PROCNO 1

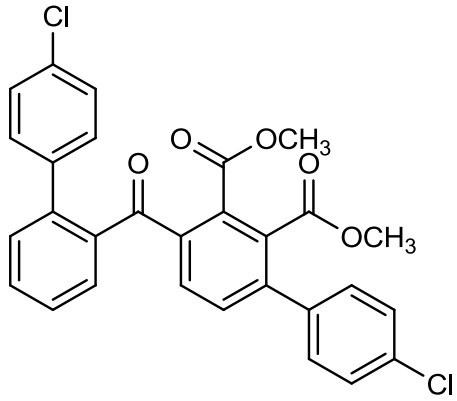
F2 - Acquisition Parameters
Date_ 20110103
Time 20.13
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 4
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 322.5
DW 81.000 usec
DE 6.00 usec
TE 297.0 K
D1 1.00000000 sec
TD0 1

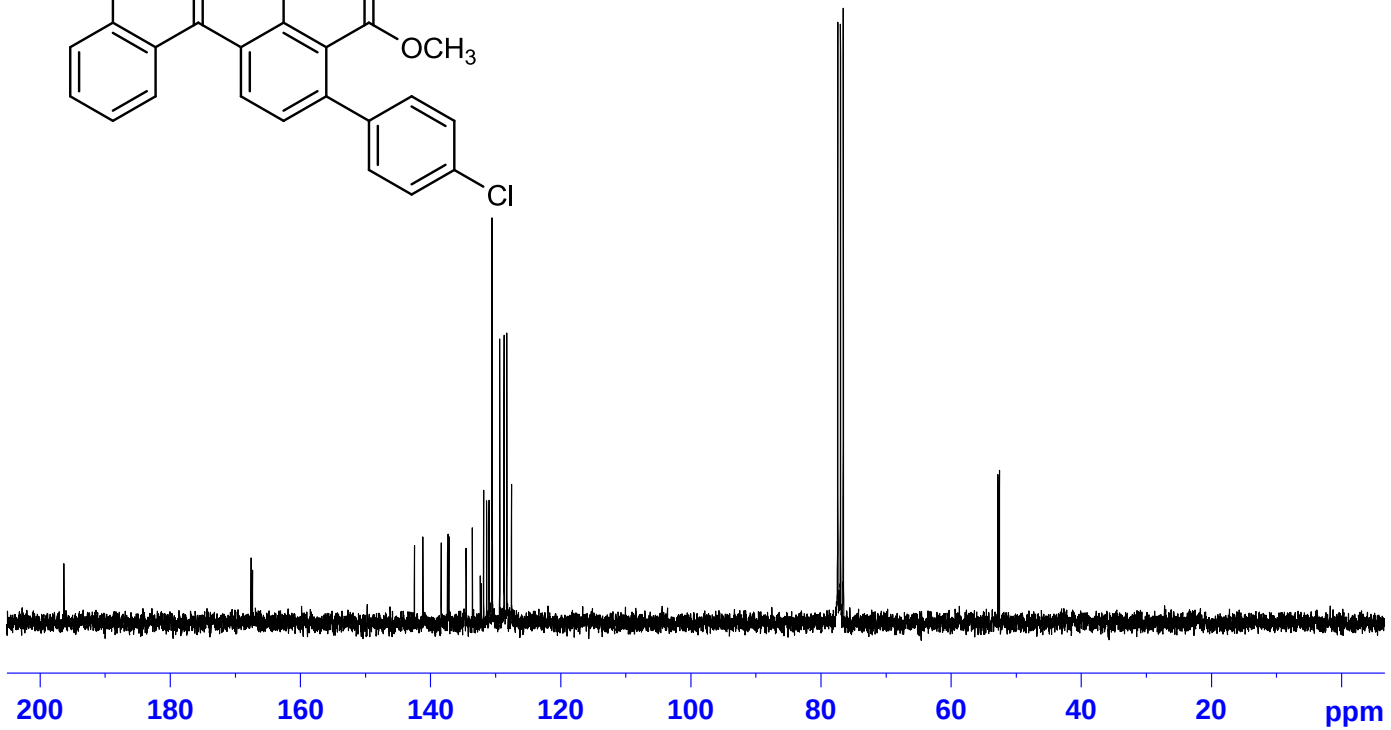
======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 0.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300375 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

Bunescu AB 365
Au13C CDCl3 /opt/topspin 1101 16

196.37
167.59
167.38
142.49
141.21
138.37
137.41
137.32
137.19
134.53
133.59
132.36
132.24
131.86
131.38
131.03
130.58
130.52
129.38
128.71
128.30
127.57
52.82
52.58





BRUKER

Current Data Parameters
NAME AB365
EXPNO 11
PROCNO 1

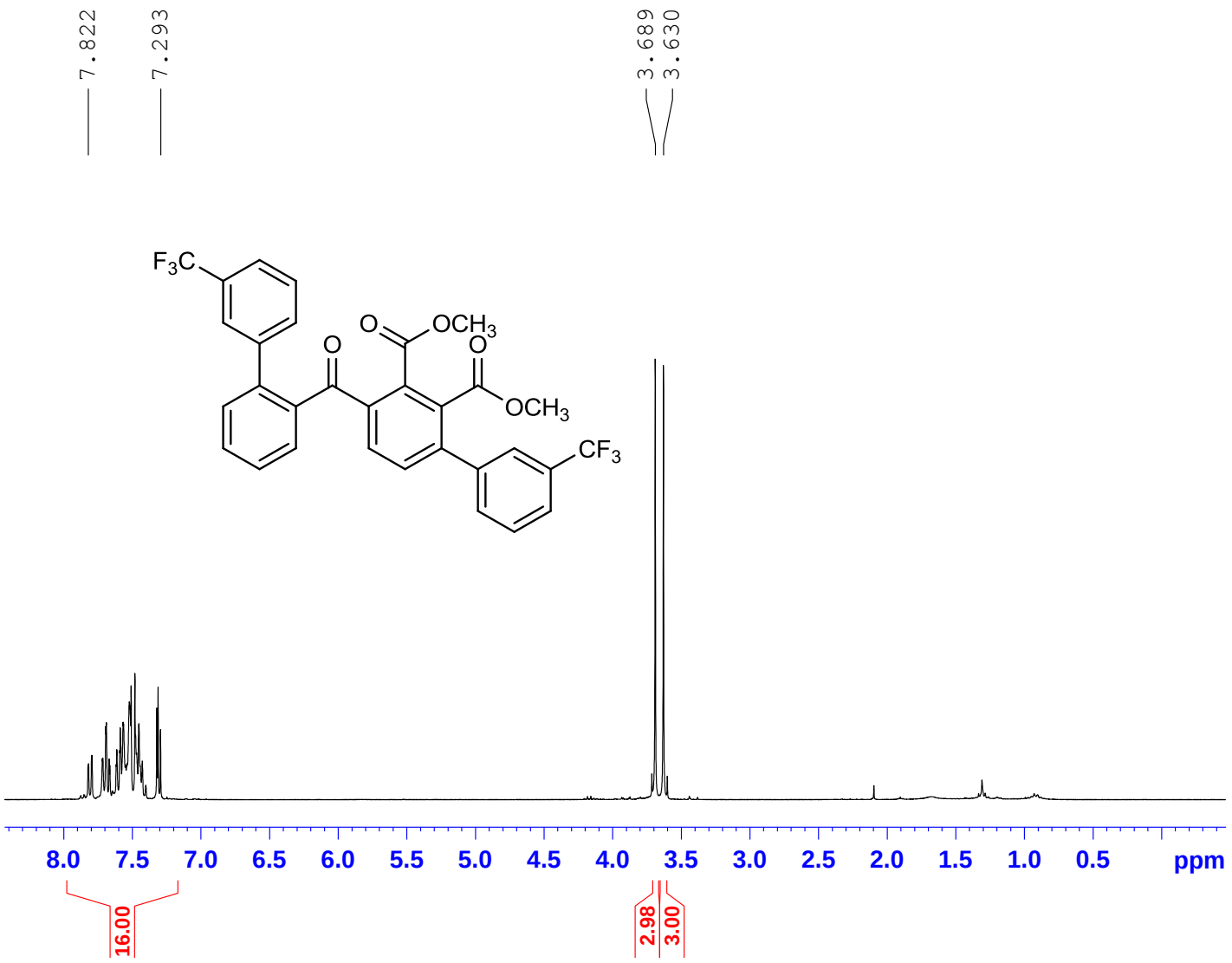
F2 - Acquisition Parameters
Date_ 20110103
Time 20.25
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 256
DS 4
SWH 21097.047 Hz
FIDRES 0.643831 Hz
AQ 0.7766516 sec
RG 32768
DW 23.700 usec
DE 6.00 usec
TE 297.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.60 usec
PL1 -1.10 dB
SFO1 75.4771825 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 19.00 dB
PL13 21.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677551 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

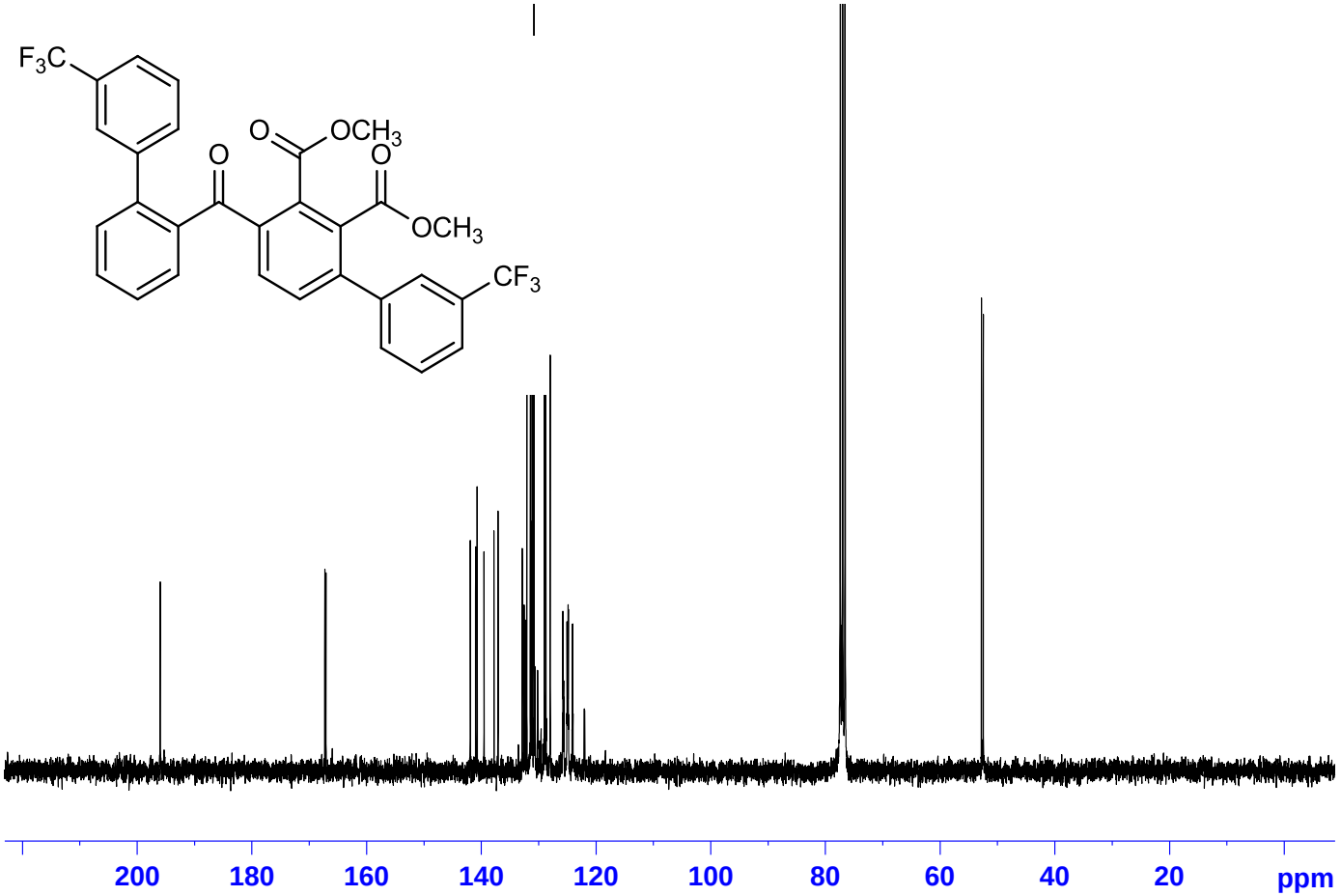
Bunescu AB 366
Au1H CDCl3 /opt/topspin 1101 17




```
Current Data Parameters
NAME                  AB366
EXPNO                    10
PROCNO                    1

F2 - Acquisition Parameters
Date_              20110103
Time                  20.44
INSTRUM               AV300
PROBHD    5 mm PABBO BB-
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       32
DS                        4
SWH                6172.839 Hz
FIDRES             0.188380 Hz
AQ                 2.6542580 sec
RG                     322.5
DW                  81.000 usec
DE                    6.00 usec
TE                   297.0 K
D1              1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     1H
P1                   11.00 usec
PL1                   0.00 dB
SFO1          300.1318534 MHz

F2 - Processing parameters
SI                    32768
SF            300.1300000 MHz
WDW                      EM
SSB                       0
LB                    0.10 Hz
GB                        0
PC                    1.00
```

Bunescu AB 366
Au13C CDCl3 /opt/topspin 1101 25

196.01 167.31 167.10 141.93 141.00 140.73 139.54 137.80 137.10 132.87 132.54 132.32 132.07 131.46 131.30 131.15 130.83 130.69 130.63 130.20 129.60 129.01 128.75 128.00 125.79 125.74 125.66 125.05 125.00 124.94 124.89 124.84 124.13 124.08 52.82 52.49



Current Data Parameters
NAME AB366
EXPNO 13
PROCNO 1

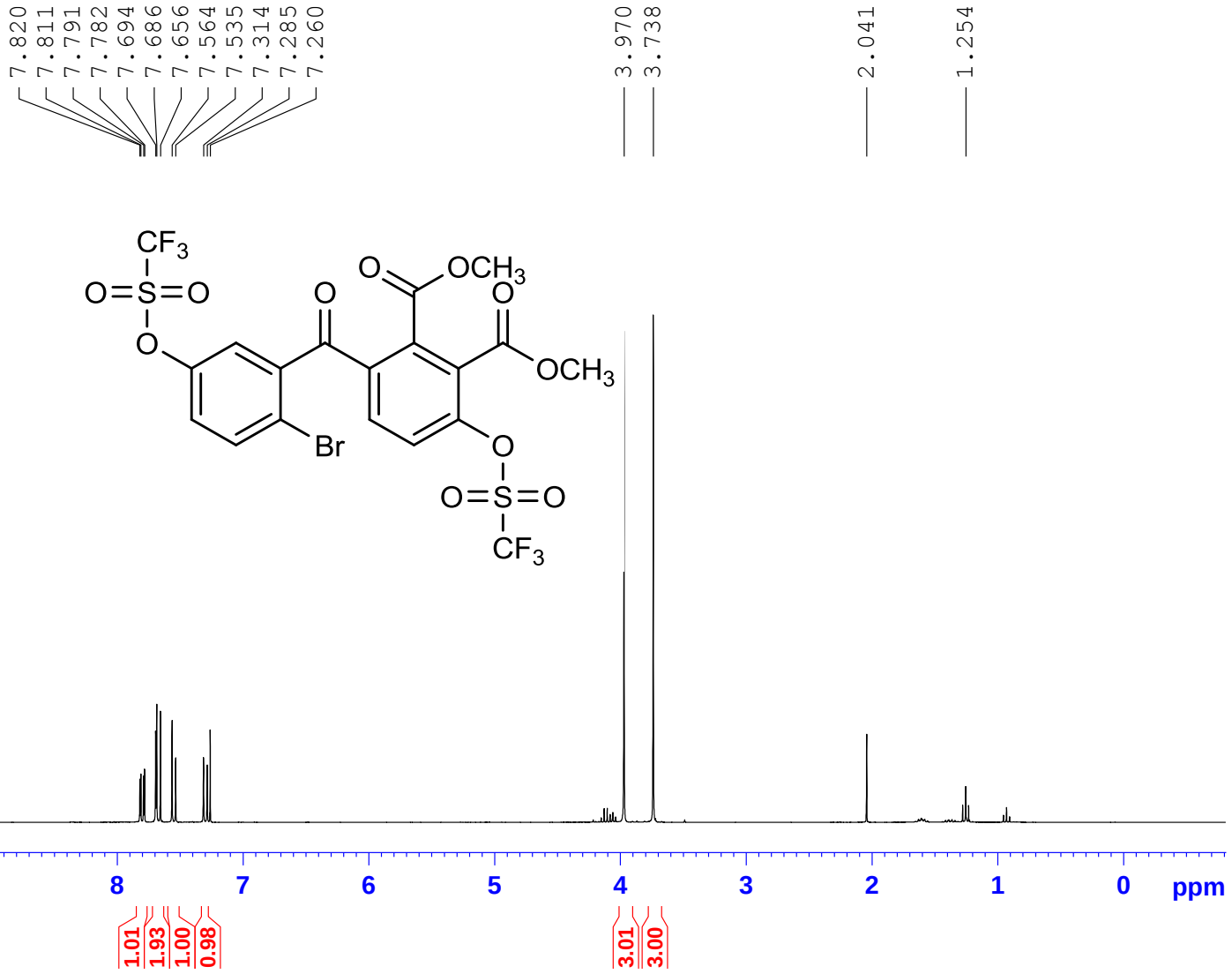
F2 - Acquisition Parameters
Date_ 20110105
Time 1.08
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 4000
DS 4
SWH 21097.047 Hz
FIDRES 0.643831 Hz
AQ 0.7766516 sec
RG 32768
DW 23.700 usec
DE 6.00 usec
TE 297.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.60 usec
PL1 -1.10 dB
SFO1 75.4771825 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 19.00 dB
PL13 21.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677536 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Bunescu AB389
Au1H CDCl3 /opt/topspin 1103 43



Current Data Parameters
NAME AB389 1H
EXPNO 10
PROCNO 1

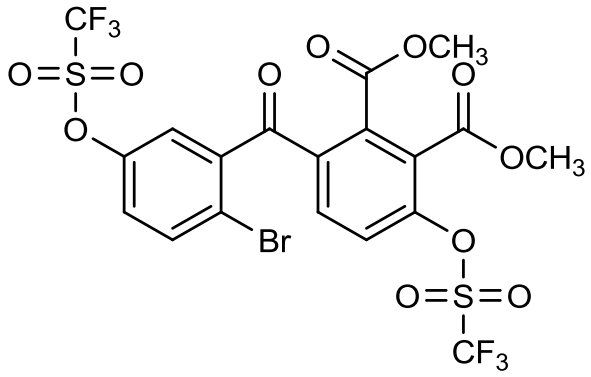
F2 - Acquisition Parameters
Date_ 20110309
Time 5.19
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 4
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 322.5
DW 81.000 usec
DE 6.00 usec
TE 297.0 K
D1 1.00000000 sec
TD0 1

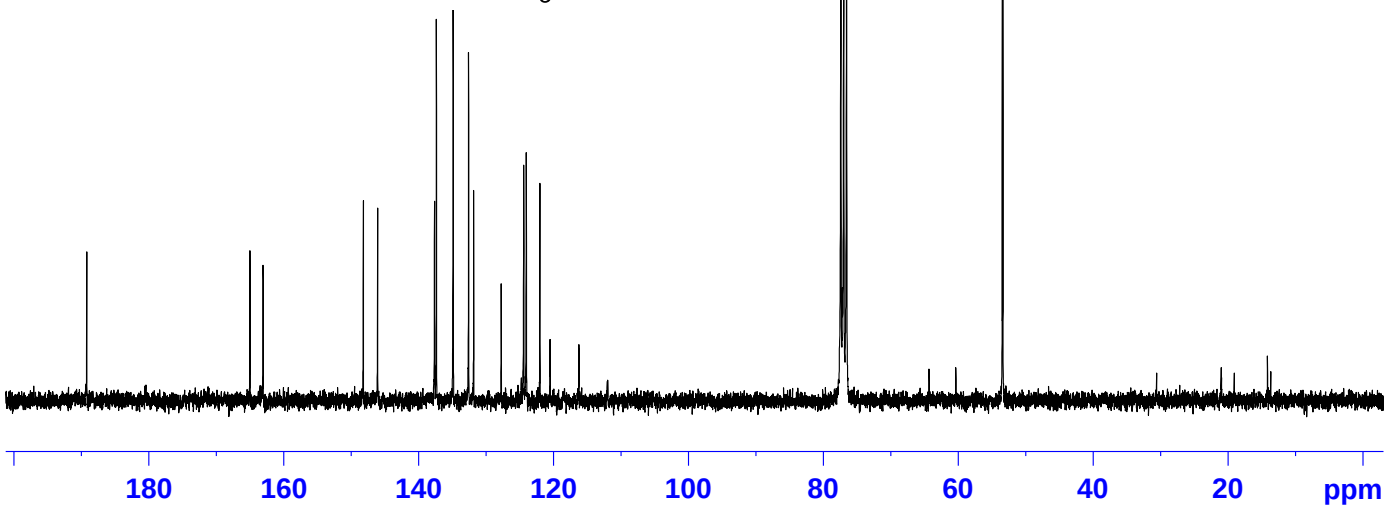
======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 0.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300151 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

Bunescu AB389
Au13C CDCl3 /opt/topspin 1103 34

189.22
165.02
163.07
148.21
146.09
137.66
137.37
134.88
132.60
131.85
127.76
124.79
124.43
124.05
122.03
120.54
120.49
116.29
116.24
111.98
53.47
53.39





Current Data Parameters
NAME AB389 13C
EXPNO 10
PROCNO 1

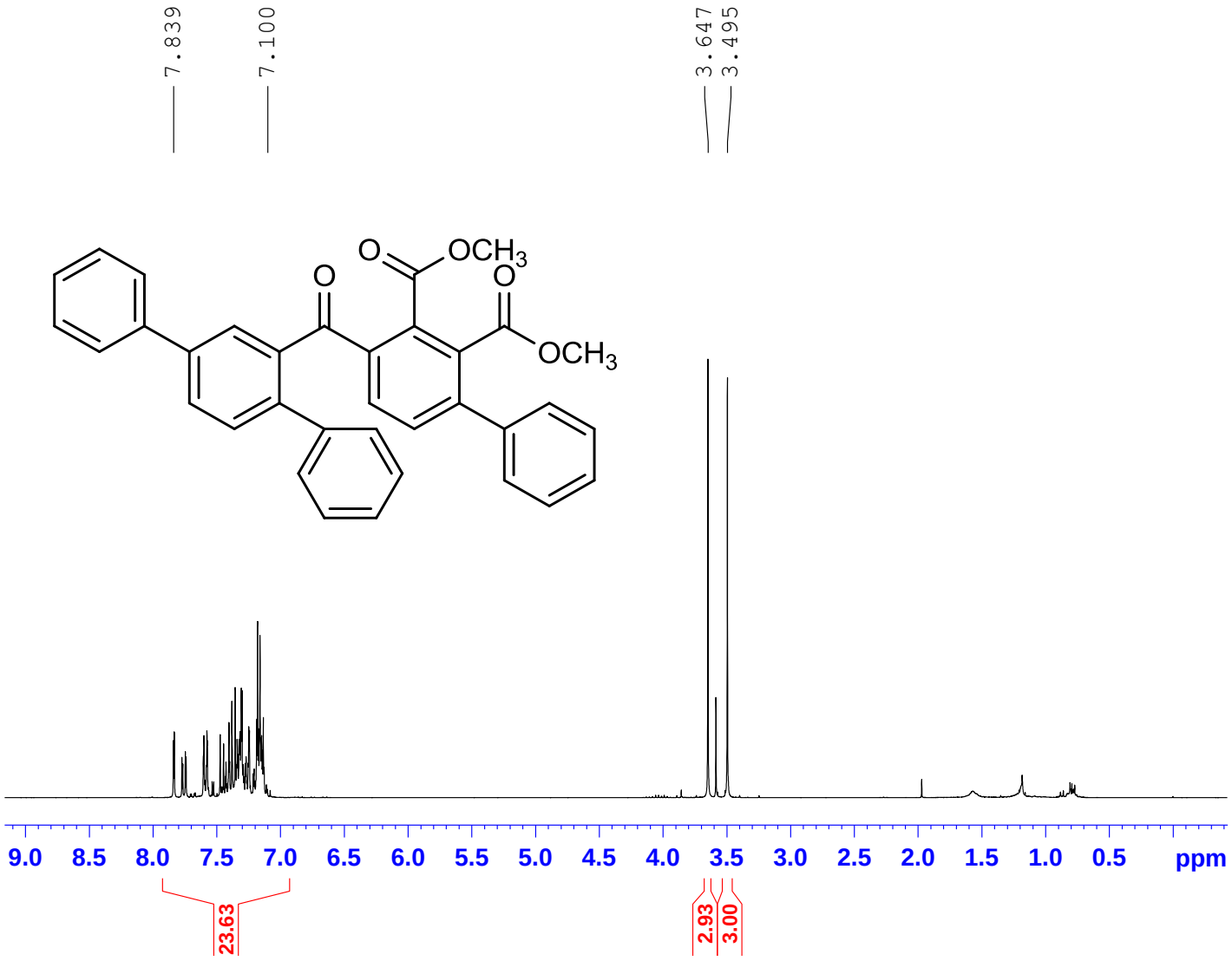
F2 - Acquisition Parameters
Date_ 20110312
Time 7.39
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 4000
DS 4
SWH 21097.047 Hz
FIDRES 0.643831 Hz
AQ 0.7766516 sec
RG 32768
DW 23.700 usec
DE 6.00 usec
TE 294.7 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.60 usec
PL1 -1.10 dB
SFO1 75.4771825 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 19.00 dB
PL13 21.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677553 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Bunescu AB394
Au1H CDCl3 /opt/topspin 1104 46



Current Data Parameters
NAME AB394 1H
EXPNO 10
PROCNO 1

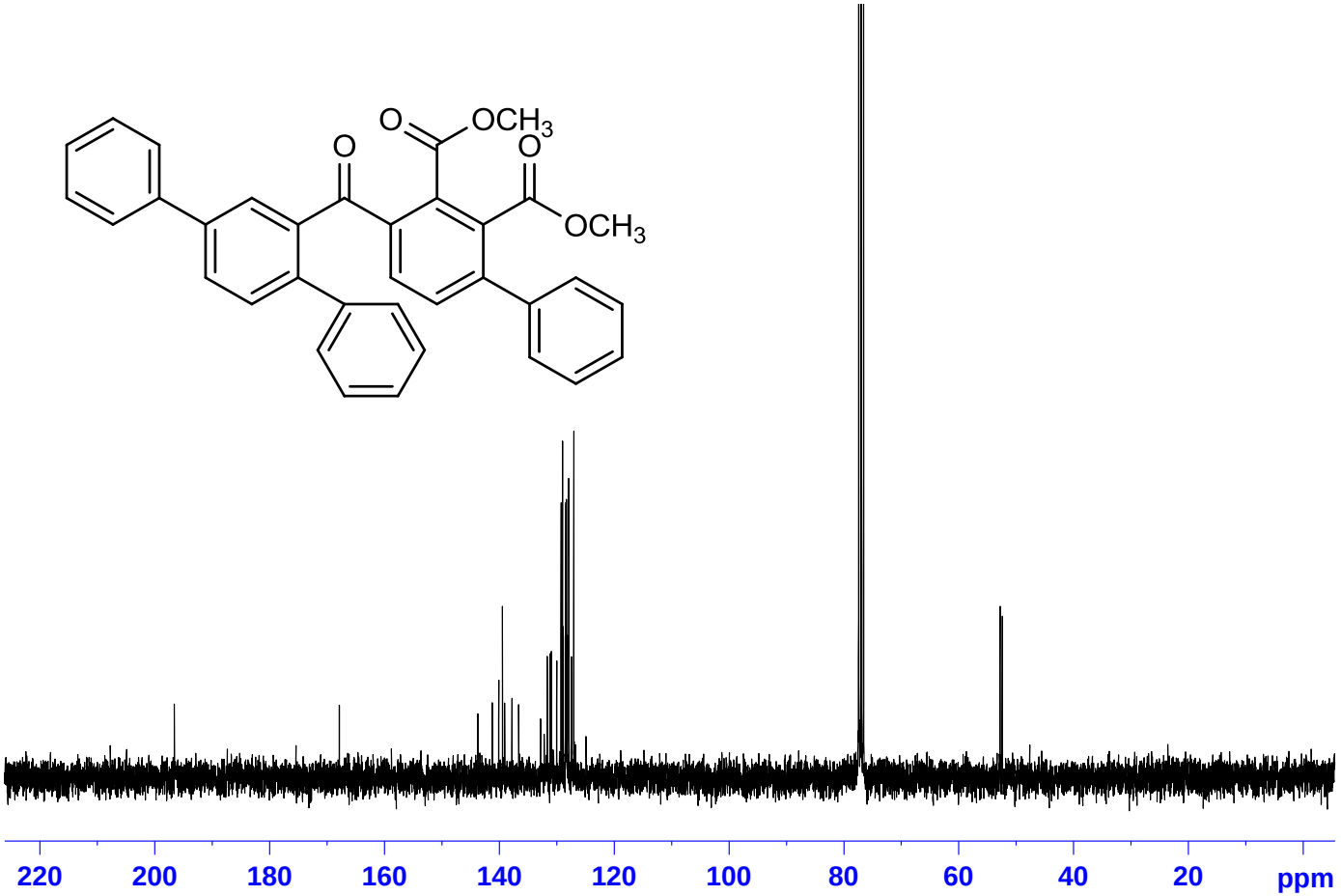
F2 - Acquisition Parameters
Date_ 20110402
Time 12.50
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 4
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 322.5
DW 81.000 usec
DE 6.00 usec
TE 294.4 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 0.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300393 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

Bunescu AB394
Au13C CDCl3 /opt/topspin 1104 46

196.61 167.89 143.76 141.24 140.12 139.46 139.07 137.82 136.63 132.84 131.66 131.15 130.95 130.02 129.25 129.00 128.87 128.43 128.31 128.20 127.93 127.46 127.05 52.81 52.42



Current Data Parameters
NAME AB394 13C
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110402
Time 13.03
INSTRUM AV300
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 256
DS 4
SWH 21097.047 Hz
FIDRES 0.643831 Hz
AQ 0.7766516 sec
RG 32768
DW 23.700 usec
DE 6.00 usec
TE 294.9 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.60 usec
PL1 -1.10 dB
SFO1 75.4771825 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 19.00 dB
PL13 21.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677545 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

(D) UV-Study

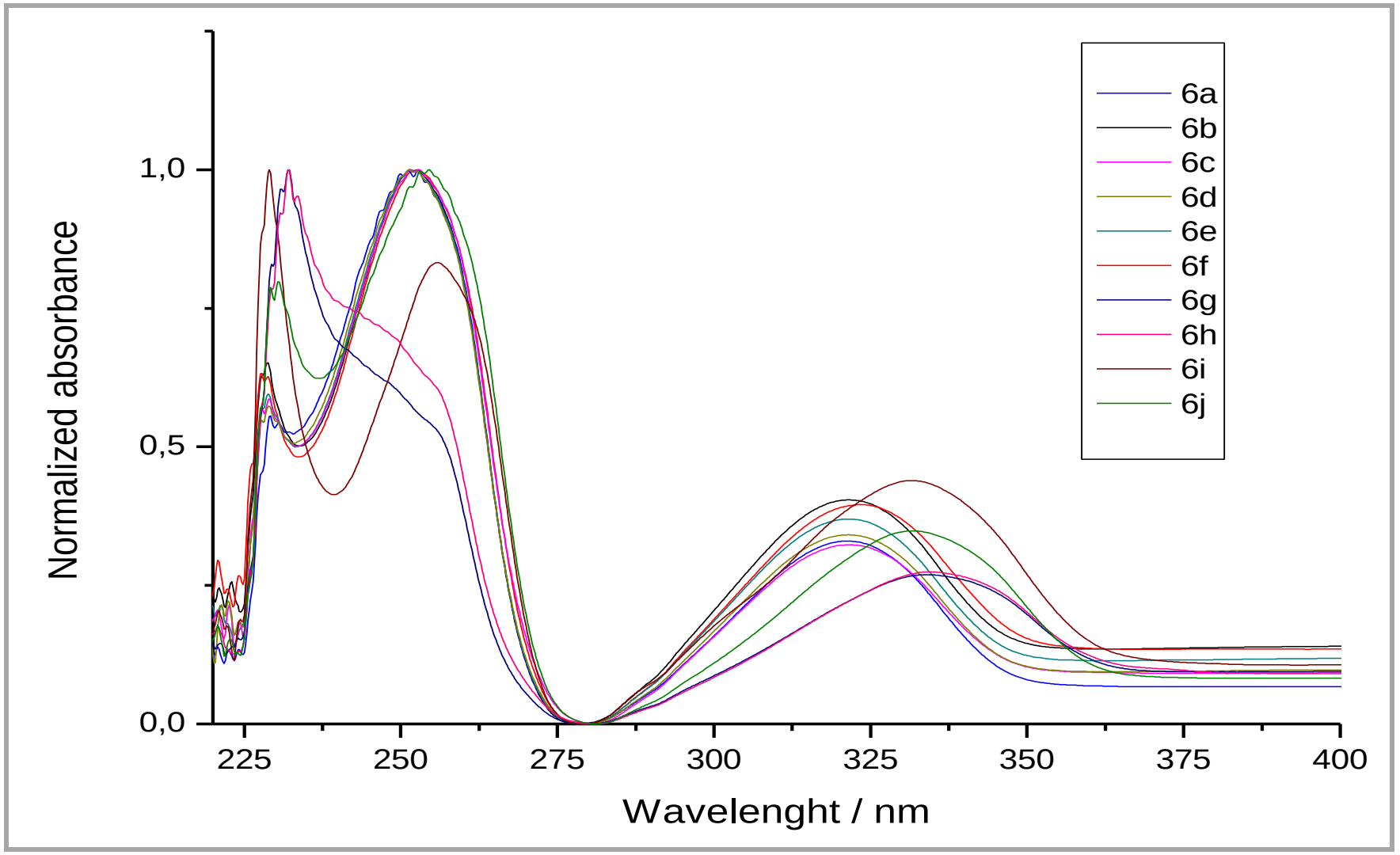


Figure 1: Absorbance spectra of **6** in DCM.

Table 2 The UV properties of **6**.

Compound	$\lambda_{max,1}$ (nm)	E_1	ε_1 ($cm^{-1}mol^{-1}l$)	$\log\varepsilon_1$	$\lambda_{max,2}$ (nm)	E_2	ε_2 ($cm^{-1}mol^{-1}l$)	$\log\varepsilon_2$	$\lambda_{max,3}$ (nm)	E_3	ε_3 ($cm^{-1}mol^{-1}l$)	$\log\varepsilon_3$
6a	229	1,10	10976	4,04	252	2,10	21016	4,32	321	0,59	5862	3,77
6b	228	0,70	6994	3,84	251	1,19	11862	4,07	321	0,35	3513	3,55
6c	228	0,85	8476	3,93	252	1,56	15608	4,19	321	0,39	3929	3,59
6d	228	0,87	8673	3,94	251	1,66	16576	4,22	321	0,44	4351	3,64
6e	228	0,74	7428	3,87	251	1,39	13890	4,14	321	0,38	3822	3,58
6f	227	0,66	6553	3,82	252	1,14	11428	4,06	323	0,34	3388	3,53
6g	-	-	-	-	232	1,87	18671	4,27	334	0,35	3491	3,54
6h	-	-	-	-	232	2,00	19982	4,30	334	0,39	3879	3,59
6i	229	1,00	9993	4,00	255	0,82	8151	3,91	331	0,38	3821	3,58
6j	230	1,45	14496	4,16	254	1,86	18614	4,27	331	0,53	5311	3,73

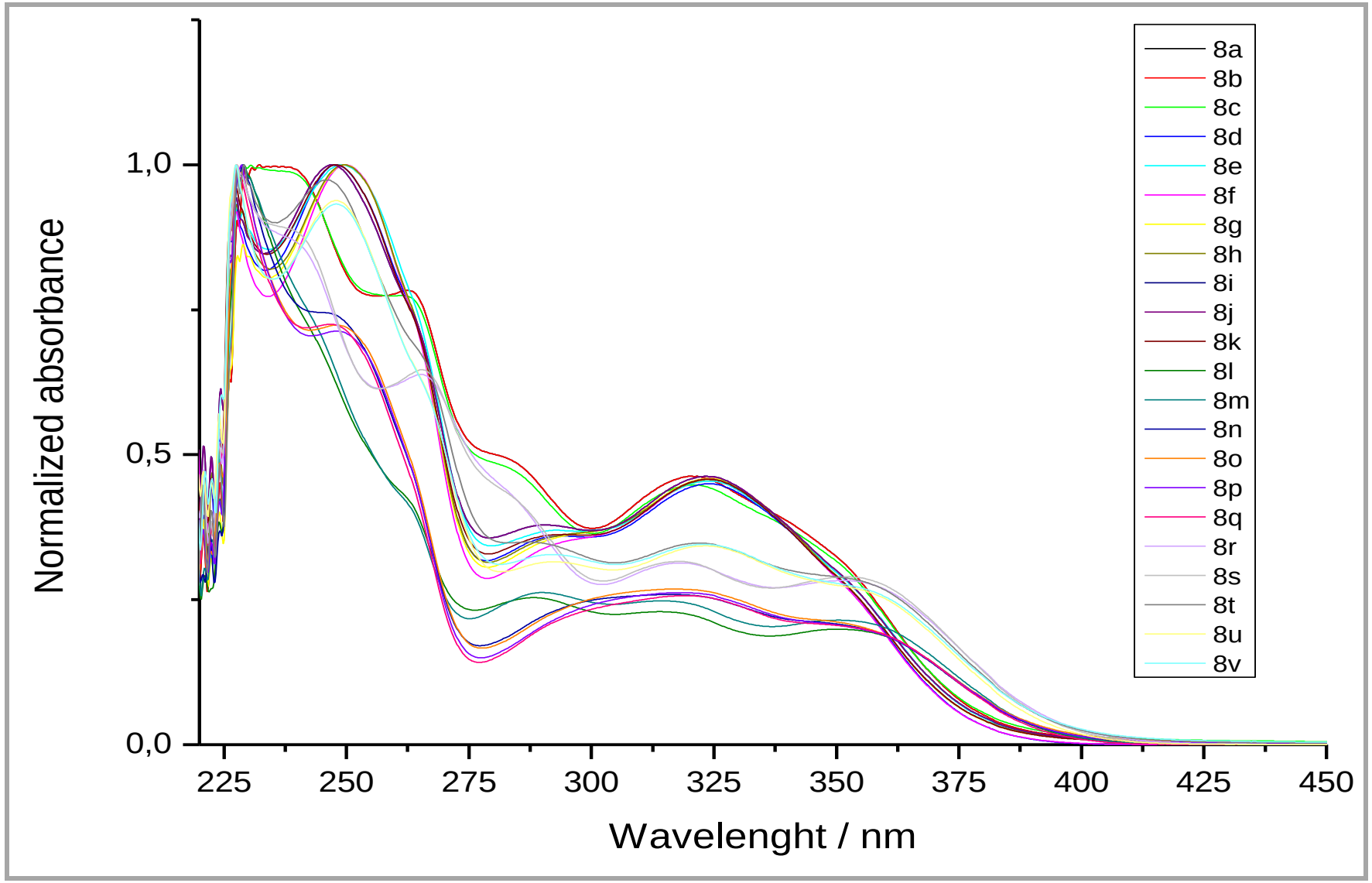


Figure 2: Absorbance spectra of **8** in DCM.

Table 3: The UV properties of **8**.

Compound	$\lambda_{max,1}$ (nm)	E_1	ε_1 ($cm^{-1}mol^{-1}l$)	$\log\varepsilon_1$	$\lambda_{max,2}$ (nm)	E_2	ε_2 ($cm^{-1}mol^{-1}l$)	$\log\varepsilon_2$	$\lambda_{max,3}$ (nm)	E_3	ε_3 ($cm^{-1}mol^{-1}l$)	$\log\varepsilon_3$
8a	229	1,25	22644	4,35	262	0,99	18013	4,26	321	0,56	10267	4,01
8b	232	1,83	18290	4,26	262	1,43	14336	4,16	320	0,85	8470	3,93
8c	229	1,18	11821	4,07	-	-	-	-	321	0,53	5311	3,73
8d	227	0,92	16733	4,22	247	0,99	18082	4,26	323	0,45	8125	3,91
8e	227	0,97	17658	4,25	248	1,03	18753	4,27	323	0,48	8644	3,94
8f	227	0,87	15855	4,20	249	0,95	17240	4,24	324	0,44	7929	3,90
8g	228	1,63	16263	4,21	249	1,88	18848	4,28	323	0,87	8653	3,94
8h	227	1,10	10955	4,04	249	1,18	11765	4,07	324	0,54	5380	3,73
8i	227	1,33	13325	4,12	249	1,43	14280	4,15	324	0,65	6490	3,81
8j	227	1,16	21060	4,32	246	1,23	22322	4,35	323	0,57	10340	4,01
8k	227	0,88	16042	4,21	247	0,92	16707	4,22	324	0,42	7636	3,88
8l	229	1,34	24364	4,39	288	0,34	6182	3,79	350	0,26	4727	3,67
8m	229	1,41	25636	4,41	289	0,37	6727	3,83	350	0,30	5455	3,74
8n	229	1,41	25636	4,41	-	-	-	-	315	0,36	6545	3,82
8o	227	1,26	22909	4,36	247	0,92	16727	4,22	315	0,33	6000	3,78
8p	228	1,32	24000	4,38	247	0,94	17091	4,23	318	0,34	6182	3,79
8q	227	0,65	11818	4,07	246	0,47	8545	3,93	318	0,16	2909	3,46
8r	227	0,83	15091	4,18	265	0,53	9636	3,98	318	0,26	4727	3,67
8s	227	0,80	14545	4,16	265	0,51	9273	3,97	352	0,23	4182	3,62
8t	229	1,70	17000	4,23	246	1,66	16600	4,22	321	0,59	5900	3,77
8u	227	0,84	15273	4,18	247	0,79	14364	4,16	323	0,29	5273	3,72
8v	227	0,75	13636	4,13	247	0,70	12727	4,10	323	0,26	4727	3,67

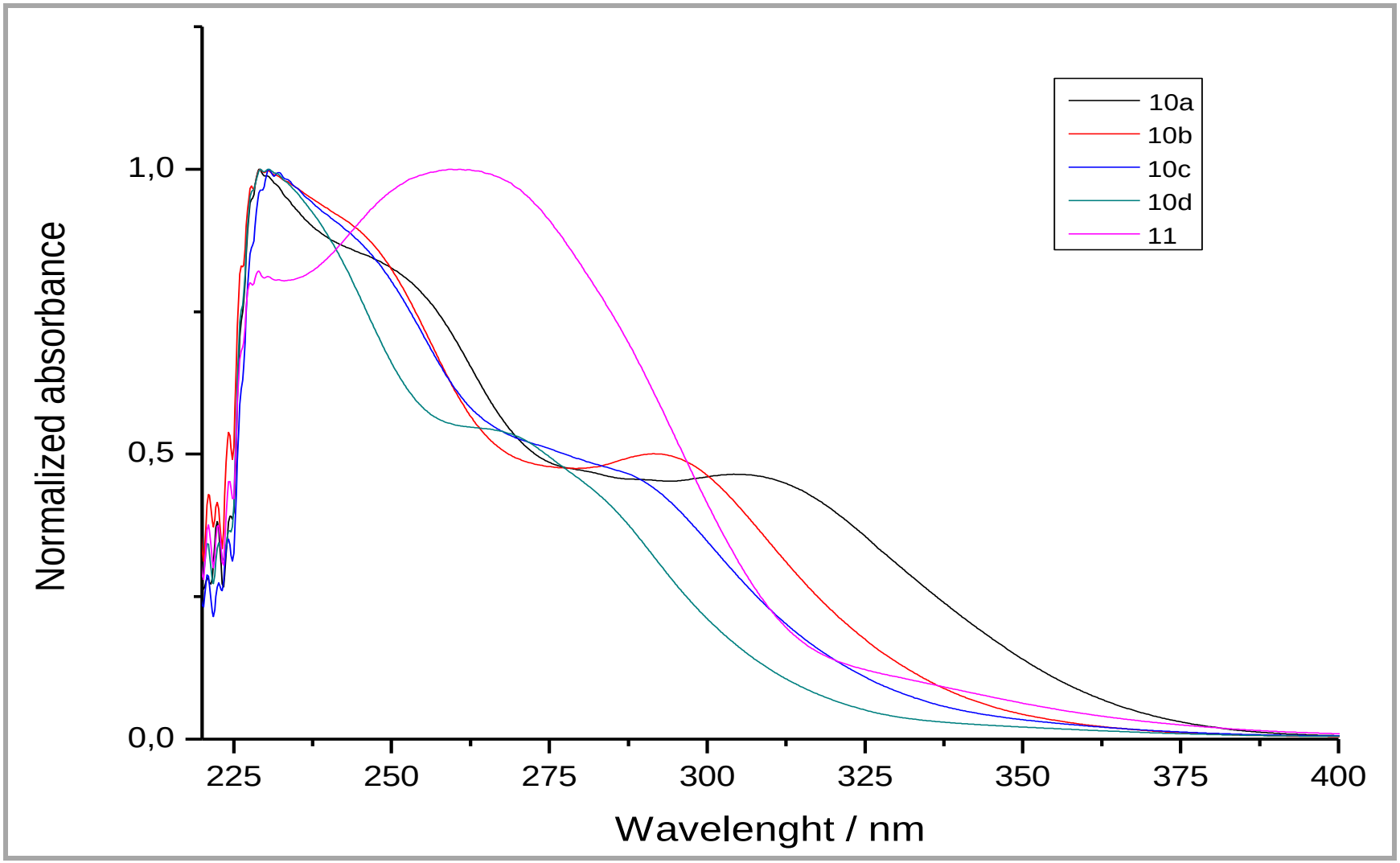


Figure 3: Absorbance spectra of **10** and **11**.

Table 4: The UV properties of **10** and **11**.

Compound	$\lambda_{max,1}$ (nm)	E_1	ε_1 ($cm^{-1}mol^{-1}l$)	$\log\varepsilon_1$	$\lambda_{max,2}$ (nm)	E_2	ε_2 ($cm^{-1}mol^{-1}l$)	$\log\varepsilon_2$
10a	229	1,52	27636	4,44	304	0,70	12727	4,10
10b	229	1,44	26182	4,42	291	0,72	13091	4,12
10c	230	2,03	36909	4,57	-	-	-	-
10d	230	1,38	25091	4,40	-	-	-	-
11	228	1,26	28000	4,45	259	1,54	34222	4,53

(E) X-Ray structure of compounds

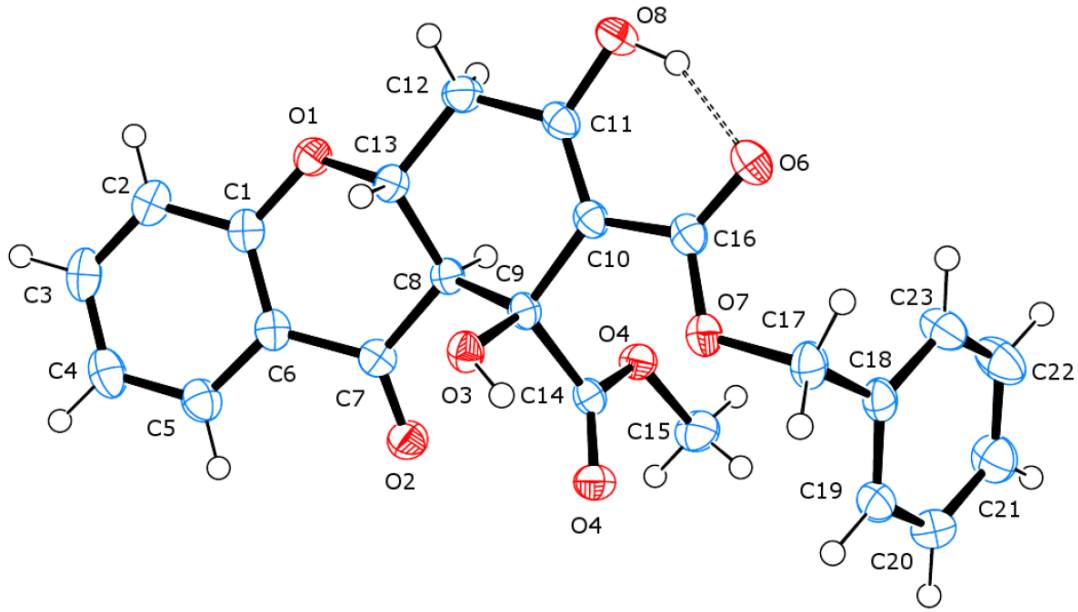


Figure 4. Molecular structure of compound **6c**

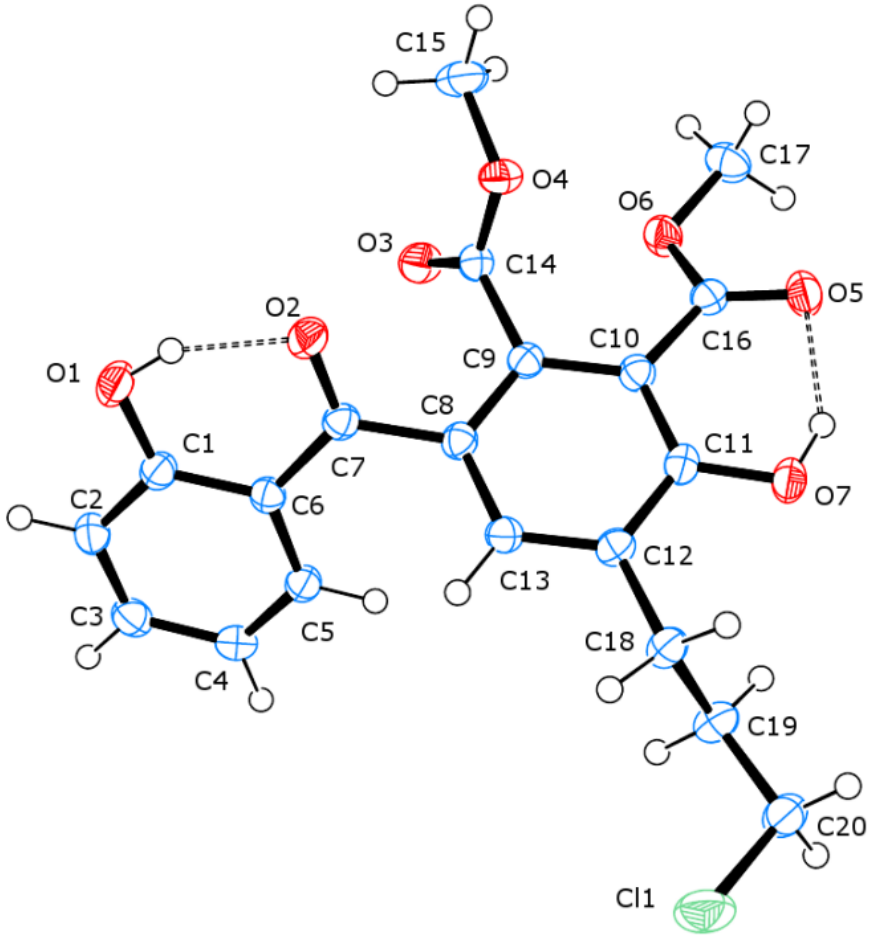


Figure 5. Molecular structure of compound **8j**

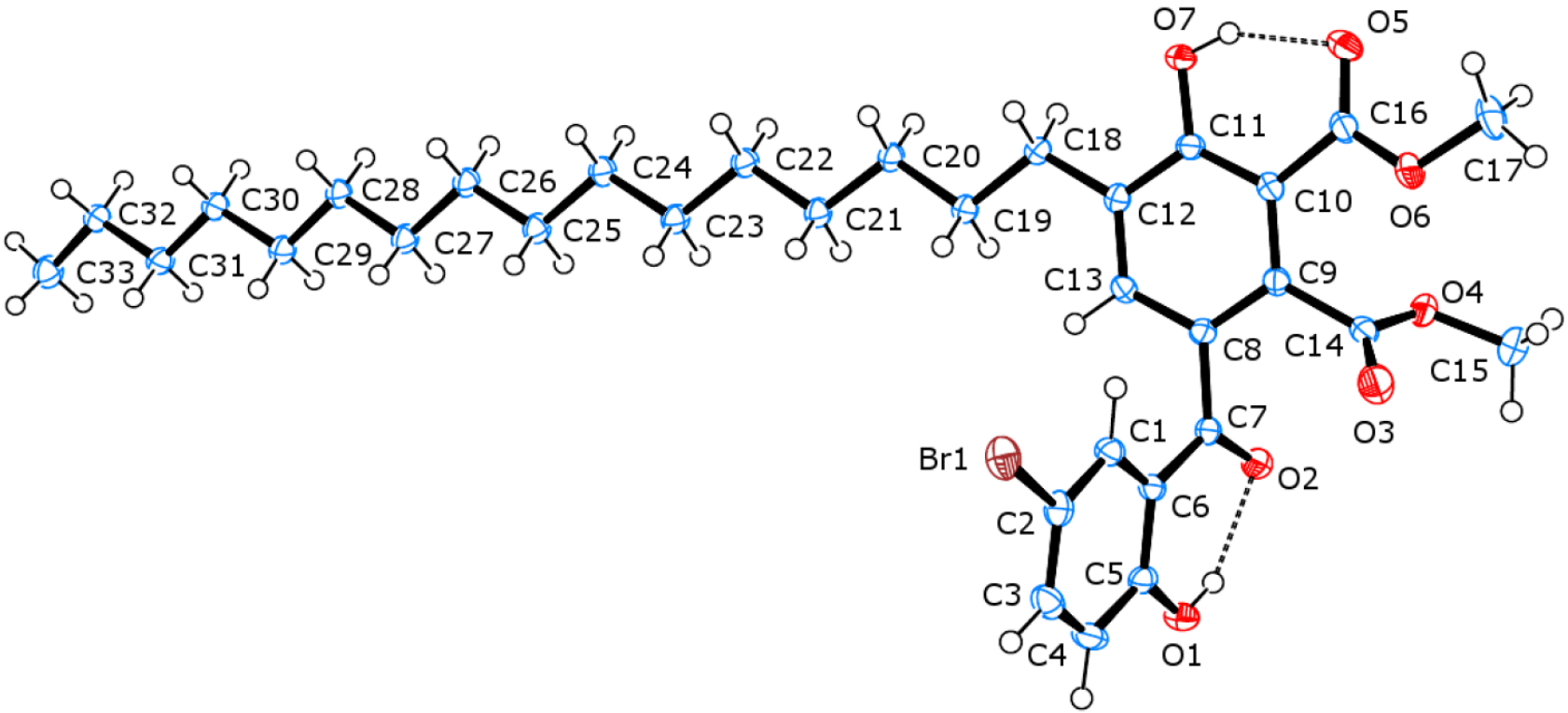


Figure 6. Molecular structure of compound **8p**