

β -Cyclodextrin/IBX in water: Highly facile biomimetic one pot deprotection of THP/MOM/Ac/Ts ethers and concomitant oxidative cleavage of chalcone epoxides and oxidative dehydrogenation of alcohols

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1. General methods

Unless otherwise noted, chemicals were purchased from commercial suppliers at the highest purity grade available and were used without further purification. Solvents were distilled by standard methods. Thin layer chromatography was performed on Merck precoated 0.25 mm silica gel plates (60F-254) using UV light as visualizing agent and/or iodine as developing agent. Silica gel (100-200 mesh) was used for column chromatography. IR spectra were recorded on FT-IR spectrometer and expressed as wave numbers (cm^{-1}). ^1H and ^{13}C NMR spectra were recorded on a Brüker (500 MHz & 125 MHz) & Jeol (400 MHz & 100 MHz) spectrometer. Spectra were referenced internally to the residual proton resonance in CDCl_3 (δ 7.26 ppm) or with tetramethylsilane (TMS, δ 0.00 ppm) as the internal standard. Chemical shifts (δ) were reported as part per million (ppm) in δ scale downfield from TMS. ^{13}C NMR spectra were referenced to CDCl_3 (δ 77.23 ppm, the middle peak). Coupling constants are expressed in Hz. The following abbreviations are used to explain the multiplicities: s = singlet, d = doublet, t = triplet, dd = doublet of doublets, m = multiplet, br = broad. High-resolution mass spectra (HRMS) were obtained on a Brüker micrOTOF™-Q II mass spectrometer (ESIMS).

2. (a) General procedure for deprotection of THP/MOM/Ac/Ts ethers and concomitant oxidative cleavage of chalcone epoxides (1a-11a). The chalcone epoxides (1 mmol) dissolved in water (2 mL) and 3-4 drops of acetone was added to an aqueous solution of β -cyclodextrin (1 mmol in 10 mL of water) at 60 °C and allowed to cool to room temperature. Then, IBX (1.1 or 2.2 mmol depending on the desired product) was added while stirring and stirring was continued for 40-60 min at 60 °C. After completion of reaction, the mixture was cooled to room temperature and extracted with EtOAc (3 \times 15 mL), dried, and concentrated in a vacuo. The crude product was purified by silica gel column chromatography using

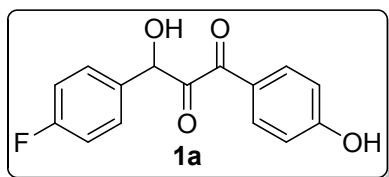
hexane/ethyl acetate (8:2) as an eluent if required otherwise compounds were pure enough for the spectral elucidation.

(b) General procedure for deprotection of THP/MOM/Ac/Ts ethers and concomitant oxidative dehydrogenation of alcohols (15a-28a). To a solution of alcohol (15-28) in water (2 mL) and 3-4 drops of acetone was added to an aqueous solution of β -cyclodextrin (1 mmol in 10 mL of water) at 60 °C and allowed to cool to room temperature. Then, IBX (1.5 equiv. per alcohol or C-C bond to be oxidized) was added while stirring. The mixture was heated to 60 °C, and the reaction was constantly monitored by TLC until complete consumption of starting material was observed. The reaction mixture was cooled to room temperature and extracted with EtOAc (3 \times 15 mL). The organic layer was washed with 5% aq. NaHCO₃ and dried over anhydrous Na₂SO₄ and concentrated in a vacuo. The crude product was purified by silica gel column chromatography using hexane/ethyl acetate (8:2) as an eluent if required otherwise compounds were pure enough for the spectral elucidation.

(c) General procedure for deprotection of THP/MOM/Ac/Ts ethers (29a-38a). β -cyclodextrin (0.1 mmol) was dissolved in water (25 ml) at 60 °C; THP/MOM/Ac/Ts ether (1 mmol) in water: acetone mixture (2 ml: 3-4 drops) was added slowly with stirring. The stirring was continued at 60 °C for the specified time (Table 5). Then the reaction mixture was cooled to room temperature and extracted with EtOAc (3 \times 15 mL), dried over anhydrous Na₂SO₄ and the solvent was removed in vacuo. The crude product was purified by silica gel column chromatography using hexane/ethyl acetate (9:1) as eluent if required otherwise compounds were pure enough for the spectral elucidation.

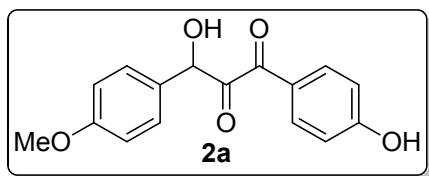
3. Characterization data for representative compounds

3-(4-fluorophenyl)-3-hydroxy-1-(4-hydroxyphenyl)propane-1,2-dione (**1a**)



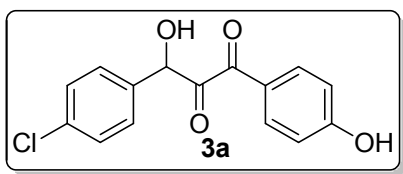
^1H NMR (400 MHz, CDCl_3 , ppm): δ 7.95-7.90 (m, 2H), 7.74 (dd, $J = 2, 10$ Hz, 2H), 7.08-7.04 (m, 2H), 7.00 (dd, $J = 2, 10$ Hz, 2H), 5.37 (s, 1H), 4.55 (s, 1H, D_2O exchangeable), 2.54 (s, 1H, D_2O exchangeable). ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 197.3, 191.5, 164.6, 163.1, 133.4, 132.5, 131.1, 129.0, 116.2, 115.8, 85.2. IR (KBr, cm^{-1}): 3420, 2945, 1687, 1649. HRMS (ESIMS) for $\text{C}_{15}\text{H}_{11}\text{FNaO}_4$ ($\text{M}+\text{Na}$) $^+$ Anal. calcd. 297.0533; found 297.0531.

3-hydroxy-1-(4-hydroxyphenyl)-3-(4-methoxyphenyl)propane-1,2-dione (**2a**)



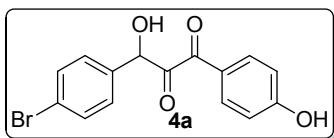
^1H NMR (400 MHz, CDCl_3 , ppm): δ 7.90 (dd, $J = 2.5, 8.5$ Hz, 2H), 7.75 (dd, $J = 2.5, 8.5$ Hz, 2H), 7.00 (dd, $J = 2.5, 8.5$ Hz, 2H), 6.88 (dd, $J = 2.5, 8.5$ Hz, 2H), 5.28 (s, 1H), 4.81 (s, 1H, D_2O exchangeable), 3.80 (s, 3H), 2.52 (s, 1H, D_2O exchangeable). ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 197.9, 191.4, 163.7, 163.2, 132.5, 130.8, 129.9, 129.0, 116.2, 113.7, 84.8, 55.5. IR (KBr, cm^{-1}): 3422, 2940, 1685, 1647. HRMS (ESIMS) for $\text{C}_{16}\text{H}_{14}\text{NaO}_5$ ($\text{M}+\text{Na}$) $^+$ Anal. calcd. 309.0733; found 309.0730.

3-(4-chlorophenyl)-3-hydroxy-1-(4-hydroxyphenyl)propane-1,2-dione (**3a**)



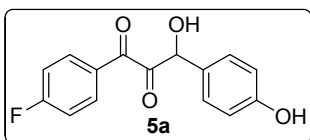
^1H NMR (400 MHz, CDCl_3 , ppm): δ 7.84 (dd, $J = 2.5, 8.5$ Hz, 2H), 7.75 (dd, $J = 2.5, 8.5$ Hz, 2H), 7.38 (dd, $J = 2.5, 8.5$ Hz, 2H), 7.00 (dd, $J = 2.5, 8.5$ Hz, 2H), 5.32 (s, 1H), 4.81 (s, 1H, D_2O exchangeable), 2.55 (s, 1H, D_2O exchangeable). ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 197.6, 191.4, 162.9, 139.7, 135.2, 132.5, 129.8, 129.1, 128.9, 116.2, 84.7. IR (KBr, cm^{-1}): 3423, 2947, 1684, 1645. HRMS (ESIMS) for $\text{C}_{15}\text{H}_{11}\text{ClNaO}_4$ ($\text{M}+\text{Na}$) $^+$ Anal. calcd. 313.0238; found 313.0232.

3-(4-bromophenyl)-3-hydroxy-1-(4-hydroxyphenyl)propane-1,2-dione (4a)



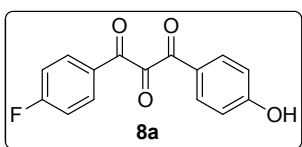
^1H NMR (400 MHz, CDCl_3 , ppm): δ 7.78-7.74 (m, 4H), 7.54 (dd, $J = 2, 11$ Hz, 2H), 6.99 (d, $J = 11$ Hz, 2H), 5.34 (s, 1H), 4.23 (s, 1H, D_2O exchangeable), 2.56 (s, 1H, D_2O exchangeable). ^{13}C NMR (125 MHz, CDCl_3 , ppm): δ 197.8, 191.5, 162.9, 135.6, 132.6, 131.9, 129.9, 129.2, 128.5, 116.1, 83.7. IR (KBr, cm^{-1}): 3423, 2947, 1685, 1642. HRMS (ESIMS) for $\text{C}_{15}\text{H}_{11}\text{BrNaO}_4$ ($\text{M}+\text{Na}$) $^+$ Anal. calcd. 356.9732; found 356.9734.

1-(4-fluorophenyl)-3-hydroxy-3-(4-hydroxyphenyl)propane-1,2-dione (5a)



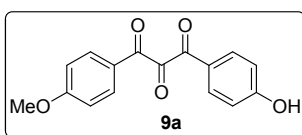
^1H NMR (400 MHz, CDCl_3 , ppm): δ 7.98-7.93 (m, 2H), 7.76 (dd, $J = 2, 10$ Hz, 2H), 7.11-7.08 (m, 2H), 6.99 (dd, $J = 2, 10$ Hz, 2H), 5.35 (s, 1H), 4.54 (s, 1H, D_2O exchangeable), 2.55 (s, 1H, D_2O exchangeable). ^{13}C NMR (125 MHz, CDCl_3 , ppm): δ 191.8, 191.2, 165.5, 162.8, 132.7, 132.6, 132.5, 125.8, 116.6, 116.4, 84.3. IR (KBr, cm^{-1}): 3420, 2946, 1686, 1647. HRMS (ESIMS) for $\text{C}_{15}\text{H}_{11}\text{FNaO}_4$ ($\text{M}+\text{Na}$) $^+$ Anal. calcd. 297.0533; found 297.0535.

1-(4-fluorophenyl)-3-(4-hydroxyphenyl)propane-1,2,3-trione (8a)



^1H NMR (400 MHz, CDCl_3 , ppm): δ 7.92-7.89 (m, 2H), 7.71 (dd, $J = 1.5, 11$ Hz, 2H), 7.06-7.02 (m, 2H), 6.97 (dd, $J = 1.5, 10.5$ Hz, 2H), 2.71 (s, 1H, D_2O exchangeable). ^{13}C NMR (125 MHz, CDCl_3 , ppm): δ 189.5, 166.9, 164.7, 133.4, 132.5, 131.0, 129.1, 116.1, 115.8. IR (KBr, cm^{-1}): 3425, 2948, 1640, 1599, 1582. HRMS (ESIMS) for $\text{C}_{15}\text{H}_9\text{FNaO}_4$ ($\text{M}+\text{Na}$) $^+$ Anal. calcd. 295.0377; found 295.0372.

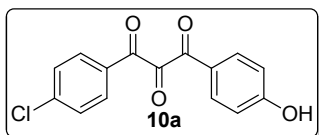
1-(4-hydroxyphenyl)-3-(4-methoxyphenyl)propane-1,2,3-trione (9a)



^1H NMR (400 MHz, CDCl_3 , ppm): δ 7.88 (dd, $J = 2.5, 9$ Hz, 2H), 7.73 (d, $J = 11$ Hz, 2H), 6.99 (d, $J = 11$ Hz, 2H), 6.86 (dd, $J = 2.5, 11$ Hz, 2H), 4.94 (s, 1H, D_2O exchangeable), 3.77 (s, 3H). ^{13}C

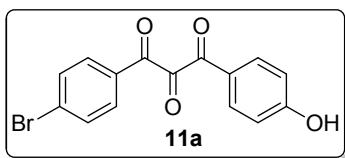
NMR (125 MHz, CDCl₃, ppm): δ 188.4, 163.7, 163.2, 132.5, 130.8, 129.9, 129.0, 116.2, 113.7, 55.4. IR (KBr, cm⁻¹): 3420, 2945, 1643, 1595, 1582. HRMS (ESIMS) for C₁₆H₁₂NaO₄ (M+Na)⁺ Anal. calcd. 307.0576; found 307.0569.

1-(4-chlorophenyl)-3-(4-hydroxyphenyl)propane-1,2,3-trione (10a)



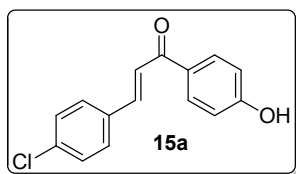
¹H NMR (400 MHz, CDCl₃, ppm): δ 7.84 (dd, J = 2.5, 8.5 Hz, 2H), 7.75 (dd, J = 2.5, 8.5 Hz, 2H), 7.38 (dd, J = 2.5, 8.5 Hz, 2H), 7.00 (dd, J = 2.5, 8.5 Hz, 2H), 4.81 (s, 1H, D₂O exchangeable). ¹³C NMR (125 MHz, CDCl₃, ppm): δ 189.7, 162.9, 139.7, 135.2, 132.5, 129.8, 129.2, 128.9, 116.1. IR (KBr, cm⁻¹): 3421, 2946, 1644, 1585. HRMS (ESIMS) for C₁₅H₉ClNaO₄ (M+Na)⁺ Anal. calcd. 311.0081; found 311.0080.

1-(4-bromophenyl)-3-(4-hydroxyphenyl)propane-1,2,3-trione (11a)



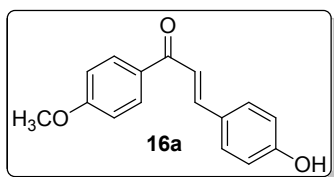
¹H NMR (400 MHz, CDCl₃, ppm): δ 7.83 (dd, J = 2.5, 10.5 Hz, 2H), 7.74 (dd, J = 2.5, 10.5 Hz, 2H), 7.37 (dd, J = 2.5, 10.5 Hz, 2H), 6.98 (d, J = 10.5 Hz, 2H), 4.94 (s, 1H, D₂O exchangeable). ¹³C NMR (125 MHz, CDCl₃, ppm): δ 189.1, 162.8, 135.6, 132.6, 131.9, 129.9, 129.2, 128.5, 116.1. IR (KBr, cm⁻¹): 3421, 2946, 1644, 1585. HRMS (ESIMS) for C₁₅H₉BrNaO₄ (M+Na)⁺ Anal. calcd. 354.9576; found 354.9571.

(E)-3-(4-chlorophenyl)-1-(4-hydroxyphenyl)prop-2-en-1-one (15a)



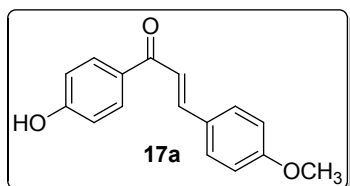
¹H NMR (CDCl₃, 500 MHz, ppm) δ 7.99 (d, J = 8 Hz, 2H), 7.77 (d, J = 15.5 Hz, 1H), 7.63 (t, J = 8 Hz, 2H), 7.46 (d, J = 15.5 Hz, 1H), 7.10 (t, J = 8.5 Hz, 2H), 6.95 (d, J = 8 Hz, 2H), 5.38 (s, 1H, D₂O exchangeable). ¹³C NMR (CDCl₃, 125 MHz, ppm) δ 186.8, 162.0, 141.3, 131.4, 130.9, 130.8, 128.9, 121.8, 115.8, 115.2. IR (KBr, ν_{\max} = cm⁻¹): 3410, 2926, 2875, 1686, 1599, 1265, 1078, 862, 730. GC-MS (m/z): 302 [M⁺, C₁₅H₁₁BrO₂], 304 [M+2].

(E)-3-(4-hydroxyphenyl)-1-(4-methoxyphenyl)prop-2-en-1-one (16a)



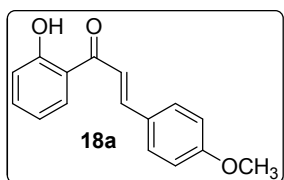
^1H NMR (CDCl_3 , 500 MHz, ppm) δ 8.03 (d, $J = 8$ Hz, 2H), 7.74 (d, $J = 15.5$ Hz, 1H), 7.56 (d, $J = 8.5$ Hz, 2H), 7.51 (d, $J = 16$ Hz, 1H), 7.38 (d, $J = 8.5$ Hz, 2H), 6.98 (d, $J = 9$ Hz, 2H), 5.48 (s, 1H, D_2O exchangeable), 3.89 (s, 3H). ^{13}C NMR (CDCl_3 , 125 MHz, ppm) δ 188.2, 163.9, 142.7, 131.4, 131.3, 130.1, 121.5, 116.8, 116.6, 114.2, 55.1. IR (KBr, $\nu_{\text{max}} = \text{cm}^{-1}$): 3410, 2928, 2880, 1684, 1599, 1265. GC-MS (m/z): 254 [M^+ , $\text{C}_{16}\text{H}_{14}\text{O}_3$].

(E)-1-(4-hydroxyphenyl)-3-(4-methoxyphenyl)prop-2-en-1-one (17a)



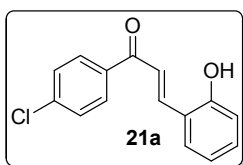
^1H NMR (CDCl_3 , 500 MHz, ppm) δ 8.03 (d, $J = 8$ Hz, 2H), 7.77 (d, $J = 16$ Hz, 1H), 7.55 (d, $J = 8$ Hz, 2H), 7.42 (d, $J = 15.5$ Hz, 1H), 6.98 (d, $J = 8$ Hz, 2H), 6.89 (d, $J = 8$ Hz, 2H), 5.82 (s, 1H, D_2O exchangeable), 3.89 (s, 3H). ^{13}C NMR (CDCl_3 , 125 MHz, ppm) δ 188.7, 163.6, 142.8, 131.2, 131.0, 130.4, 121.7, 116.3, 116.2, 114.0, 55.7. IR (KBr, $\nu_{\text{max}} = \text{cm}^{-1}$): 3410, 2926, 2875, 1686, 1599, 1265. GC-MS (m/z): 254 [M^+ , $\text{C}_{16}\text{H}_{14}\text{O}_3$].

(E)-1-(2-hydroxyphenyl)-3-(4-methoxyphenyl)prop-2-en-1-one (18a)



^1H NMR (CDCl_3 , 400 MHz, ppm) δ 7.91-7.86 (m, 2H), 7.61 (d, $J = 8.8$ Hz, 2H), 7.52 (d, $J = 15.6$ Hz, 1H), 7.49-7.45 (m, 2H), 7.00 (dd, $J = 1.2, 8.8$ Hz, 1H), 6.93 (d, $J = 8.4$ Hz, 2H), 3.84 (s, 3H), 1.68 (s, 1H, D_2O exchangeable). ^{13}C NMR (CDCl_3 , 100 MHz, ppm) δ 193.8, 163.7, 162.1, 145.5, 136.3, 130.7, 129.7, 127.4, 120.2, 118.9, 118.7, 117.7, 114.6, 55.6. IR (KBr, $\nu_{\text{max}} = \text{cm}^{-1}$): 3410, 2926, 2875, 1686, 1599, 1265. GC-MS (m/z): 254 [M^+ , $\text{C}_{16}\text{H}_{14}\text{O}_3$].

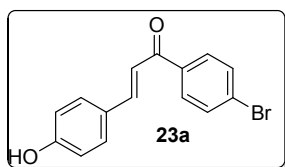
(E)-1-(4-chlorophenyl)-3-(2-hydroxyphenyl)prop-2-en-1-one (21a)



^1H NMR (CDCl_3 , 400 MHz, ppm) δ 7.92-7.84 (m, 2H), 7.64-7.58 (m, 3H), 7.53-7.49 (m, 1H), 7.41 (d, $J = 8.8$ Hz, 2H), 7.01 (d, $J = 8.4$ Hz, 1H), 6.95 (d, $J = 7.2$ Hz, 2H), 4.84 (s, 1H, D_2O exchangeable). ^{13}C

NMR (CDCl₃, 100 MHz, ppm) δ 193.6, 163.8, 144.1, 136.7, 133.2, 131.7, 130.0, 129.8, 129.5, 129.0, 120.7, 119.1, 118.9. IR (KBr, ν_{\max} = cm⁻¹): 3410, 2926, 2875, 1686, 1599, 1265. GC-MS (m/z): 258 [M⁺, C₁₅H₁₁ClO₂], 260 [M+2]⁺.

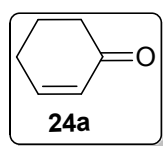
(E)-3-(4-bromophenyl)-1-(4-hydroxyphenyl)prop-2-en-1-one (23a)



¹H NMR (CDCl₃, 500 MHz, ppm) δ 7.99 (d, J = 8 Hz, 2H), 7.77 (d, J = 15.5 Hz, 1H), 7.63 (t, J = 8 Hz, 2H), 7.46 (d, J = 15.5 Hz, 1H), 7.10 (t, J = 8.5 Hz, 2H), 6.95 (d, J = 8 Hz, 2H), 5.48 (s, 1H, D₂O

exchangeable). ¹³C NMR (CDCl₃, 125 MHz, ppm) δ 186.88, 162.05, 141.32, 131.41, 130.90, 130.83, 128.92, 121.85, 115.81, 115.21. IR (KBr, ν_{\max} = cm⁻¹): 3410, 2926, 2875, 1686, 1599, 1265, 1078, 862, 730. GC-MS (m/z): 302 [M⁺, C₁₅H₁₁BrO₂], 304 [M+2].

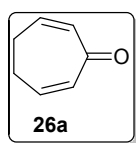
Cyclohex-2-enone (24a)



¹H NMR (CDCl₃, 500 MHz, ppm) δ 6.71-6.69 (m, 1H), 5.90 (d, J = 10 Hz, 1H), 2.35 (t, J = 1 Hz, 2H), 1.91-1.89 (m, 2H), 1.72-1.66 (m, 2H). ¹³C NMR (CDCl₃, 125 MHz, ppm) δ 198.1, 163.2, 127.1, 37.4, 24.5, 22.6. IR (KBr, ν_{\max}

= cm⁻¹): 1701, 1632. GC-MS (m/z): 96 [M⁺, C₆H₈O].

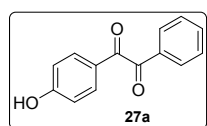
Cyclohepta-2,6-dienone (26a)



¹H NMR (CDCl₃, 500 MHz, ppm) δ 6.43 (d, J = 10.5 Hz, 2H), 6.31-6.29 (m, 2H), 2.34-2.26 (m, 4H). ¹³C NMR (CDCl₃, 125 MHz, ppm) δ 194.8, 144.2, 135.1, 28.1. IR (KBr, ν_{\max} = cm⁻¹): 1655, 1610, 1564, 1410. GC-MS (m/z): 108

[M⁺, C₇H₈O].

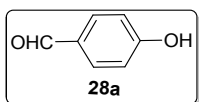
1-(4-hydroxyphenyl)-2-phenylethane-1,2-dione (27a)



¹H NMR (CDCl₃, 500 MHz, ppm) δ 7.99 (d, J = 7.5 Hz, 2H), 7.64-7.48 (m, 5H), 6.98 (d, J = 7.5 Hz, 2H), 5.48 (s, 1H, D₂O exchangeable). ¹³C

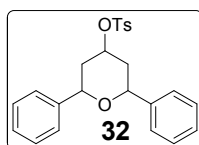
NMR (CDCl₃, 125 MHz, ppm) δ 191.7, 191.6, 162.6, 137.6, 134.7, 132.6, 130.7, 129.2, 124.7, 114.4. IR (KBr, ν_{\max} = cm⁻¹): 3410, 1686, 1640. GC-MS (m/z): 226 [M⁺, C₁₄H₁₀O₃].

4-hydroxybenzaldehyde (28a)

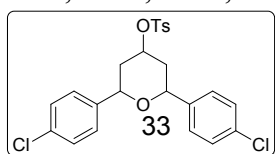


¹H NMR (CDCl₃, 500 MHz, ppm) δ 9.91 (s, 1H, D₂O exchangeable), 7.35 (d, J = 7.5 Hz, 2H), 6.94 (d, J = 8 Hz, 2H), 3.90 (s, 1H). ¹³C NMR (CDCl₃, 125 MHz, ppm) δ 192.9, 163.0, 134.7, 130.2, 117.1. IR (KBr, ν_{\max} = cm⁻¹): 3420, 2927, 2873, 1721. GC-MS (m/z): 122 [M⁺, C₇H₆O₂].

2,6-diphenyltetrahydro-2H-pyran-4-yl 4-methylbenzenesulfonate (32)



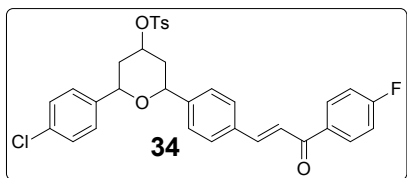
¹H NMR (500 MHz, CDCl₃, ppm): δ 7.88 (d, J = 8Hz, 2H), 7.43-7.37 (m, 10H), 7.34-7.31 (m, 2H), 5.03 (tt, J = 4.5 and 11.5Hz, 1H), 4.59 (d, J = 11.5Hz, 2H), 2.48 (s, 3H), 2.34 (dd, J = 4.5, 12.5Hz, 2H), 1.88 (q, J = 12.5Hz, 2H). ¹³C NMR (125 MHz, CDCl₃, ppm): δ 144.7, 140.9, 134.2, 129.8, 128.3, 127.7, 127.5, 125.7, 78.1, 77.4, 61.2, 39.9, 21.5. IR (KBr, cm⁻¹): 3056, 3039, 2923, 2852, 2373, 1717, 1629, 1454, 1379, 1178, 1065, 945, 903, 757, 699.



2,6-bis(4-chlorophenyl)tetrahydro-2H-pyran-4-yl 4-methylbenzenesulfonate (33)

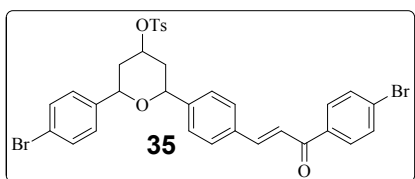
¹H NMR (500 MHz, CDCl₃, ppm): δ 7.80 (d, J = 8Hz, 2H), 7.35-7.27 (m, 10H), 4.93 (tt, J = 4.5, 11Hz, 1H), 4.50 (dd, J = 1.5, 11.5 Hz, 2H), 2.44 (s, 3H), 2.26 (dd, J = 4.5, 11.5Hz, 2H), 1.75 (q, J = 11.5Hz, 2H). ¹³C NMR (125 MHz, CDCl₃, ppm): δ 145.0, 139.3, 134.2, 133.7, 130.0, 128.7, 127.6, 127.2, 77.6, 76.8, 39.9, 21.7.

(E)-2-(4-chlorophenyl)-6-(4-(3-(4-fluorophenyl)-3-oxoprop-1-en-1-yl)phenyl)tetrahydro-2H-pyran-4-yl 4-methylbenzenesulfonate (34)



¹H NMR (500 MHz, CDCl₃, ppm): δ 8.02-8.05 (m, 4H), 7.81 (d, *J* = 8.5 Hz, 1H), 7.78 (d, *J* = 5 Hz, 1H), 7.75 (d, *J* = 5 Hz, 1H), 7.60 (dd, *J* = 8, 2.5 Hz, 4H), 7.47 (d, *J* = 15.5 Hz, 2H), 7.39 (dd, *J* = 8.5, 3 Hz, 3H), 7.33 (d, *J* = 8 Hz, 2H), 4.96 (tt, *J* = 11, 4.5 Hz, 1H), 4.56-4.49 (m, 2H), 2.43 (s, 3H), 2.25-2.33 (m, 2H), 1.76-1.83 (m, 2H). ¹³C NMR (125 MHz, CDCl₃, ppm): δ 188.8, 164.6, 146.9, 144.8, 143.4, 139.3, 134.3, 134.1, 133.8, 133.6, 131.7, 131.1, 130.0, 128.6, 128.6, 127.6, 127.2, 126.4, 121.2, 77.6, 77.3, 76.9, 39.8, 39.8, 21.7. IR (KBr, cm⁻¹): 3000, 2945, 1678, 1614, 1350, 1200, 1121, 861. HRMS (ESIMS) for C₃₃H₂₈ClFNaO₅S (M+Na)⁺ Anal. calcd. 613.1228; found 613.1220.

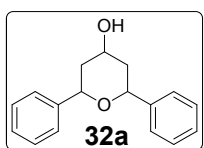
(E)-2-(4-bromophenyl)-6-(4-(3-(4-bromophenyl)-3-oxoprop-1-en-1-yl)phenyl)tetrahydro-2H-pyran-4-yl 4-methylbenzenesulfonate (35)



¹H NMR (500MHz, CDCl₃, ppm): δ 7.86 (dd, *J* = 8.5, 2Hz, 4H), 7.60 (dd, *J* = 10.5, 8.5 Hz, 6H), 7.39-7.46 (m, 6H), 7.33 (d, *J* = 8 Hz, 1H), 7.22 (d, *J* = 8.5 Hz, 1H), 4.95 (tt, *J* = 6.5, 3Hz, 1H), 4.52 (dd, *J* = 28, 10 Hz, 2H), 2.43 (s, 3H), 2.29 (dd, *J* = 24, 12.5Hz, 2H), 1.77 (q, *J* = 11 Hz, 2H). ¹³C NMR (125 MHz, CDCl₃, ppm): δ 189.18, 146.84, 143.35, 139.67, 136.75, 133.91, 133.67, 131.78, 131.46, 129.90, 128.50, 127.46, 127.42, 126.33, 121.03, 118.61, 77.43, 76.73, 72.70, 39.65, 21.55. IR (KBr, cm⁻¹): 3000, 2945, 1678, 1614, 1350, 1200, 1121, 861. HRMS (ESIMS) for C₃₃H₂₈Br₂NaO₅S (M+Na)⁺ Anal. calcd. 716.9922; found 716.9900.

(c) Spectral data of OTs deprotected product:

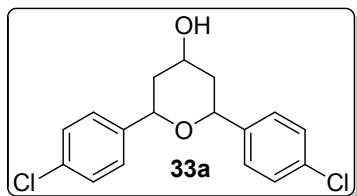
2,6-diphenyltetrahydro-2H-pyran-4-ol (32a)



¹H NMR (500 MHz, CDCl₃, ppm): δ 7.19-7.41 (m, 8H), 4.51-4.43 (m, 2H), 4.07 (tt, *J* = 4.5, 11.5 Hz, 1H), 2.28 (s, br, D₂O exchangeable, 1H, OH), 2.21 (dd, *J* = 4, 11.5Hz, 2H), 1.53 (q, *J* = 11.5 Hz, 2H). ¹³C NMR (125

MHz, CDCl₃, ppm): δ 131.4, 128.3, 127.5, 125.8, 77.8, 68.6, 42.9. IR (KBr, cm⁻¹): 3433, 2965, 2921, 2852, 1634, 1452, 1382, 1265, 1156, 1065, 900, 760, 700. GC-MS (m/z): 410 [M⁺, C₁₇H₁₈O₂].

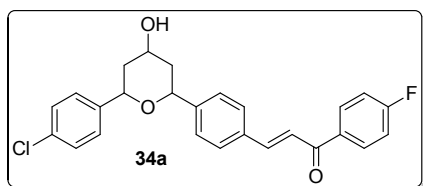
2,6-bis(4-chlorophenyl)tetrahydro-2H-pyran-4-ol (33a)



¹H NMR (500 MHz, CDCl₃, ppm): δ 7.29-7.24 (m, 8H), 4.47 (d, *J* = 11.5 Hz, 2H), 4.06 (tt, *J* = 4.5, 11.5 Hz, 1H), 2.19 (dd, *J* = 4, 11.5 Hz, 2H), 1.48 (q, *J* = 11.5 Hz, 2H). ¹³C NMR (125

MHz, CDCl₃, ppm): δ 139.2, 132.3, 127.5, 126.2, 77.8, 67.4, 41.9. IR (KBr, cm⁻¹): 3447, 2960, 2886, 1652, 1543, 1088, 804. GC-MS (m/z): 323 [M⁺, C₁₇H₁₆Cl₂O₂].

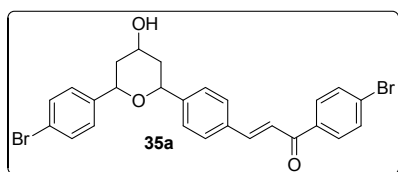
(E)-3-(4-(6-(4-chlorophenyl)-4-hydroxytetrahydro-2H-pyran-2-yl)phenyl)-1-(4-fluorophenyl)prop-2-en-1-one (34a)



¹H NMR (500 MHz, CDCl₃, ppm): δ 8.18 (d, *J* = 8 Hz, 1H), 7.95 (d, *J* = 8 Hz, 2H), 7.59 (d, *J* = 9 Hz, 3H), 7.48 (d, *J* = 8 Hz, 2H), 7.41 (d, *J* = 9 Hz, 2H), 7.36 (d, *J* = 8.5

Hz, 2H), 7.01 (d, *J* = 9 Hz, 2H), 4.66 (t, *J* = 3 Hz, 2H), 4.14 (tt, *J* = 11, 3 Hz, 1H), 2.22-2.85 (m, 2H), 2.04 (s, br, D₂O exchangeable, 1H), 1.73-1.84 (m, 2H). ¹³C NMR (125 MHz, CDCl₃, ppm): δ 188.8, 166.7, 164.6, 144.6, 143.7, 140.8, 134.5, 134.3, 131.2, 130.0, 128.7, 127.7, 126.5, 125.9, 121.6, 115.9, 78.0, 77.7, 69.4, 40.0. IR (KBr, cm⁻¹): 3434, 3010, 2922, 2843, 1734, 1626, 1456, 1256, 1069, 808.8. HRMS (ESIMS): for C₂₆H₂₂ClFNaO₃ (M+Na)⁺ Anal. calcd. 459.1139; found 459.1150.

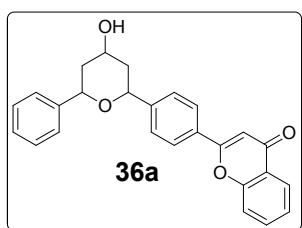
(E)-1-(4-bromophenyl)-3-(4-(6-(4-bromophenyl)-4-hydroxytetrahydro-2H-pyran-2-yl)phenyl)prop-2-en-1-one (35a)



¹H NMR (500 MHz, CDCl₃, ppm): δ 7.88 (d, *J* = 8 Hz, 2H), 7.82 (d, *J* = 8.5 Hz, 2H), 7.78 (s, 1H), 7.61-7.66 (m, 4H), 7.47 (s, 1H), 7.35 (d, *J* = 8 Hz, 2H), 7.31-7.32 (m, 2H), 4.45

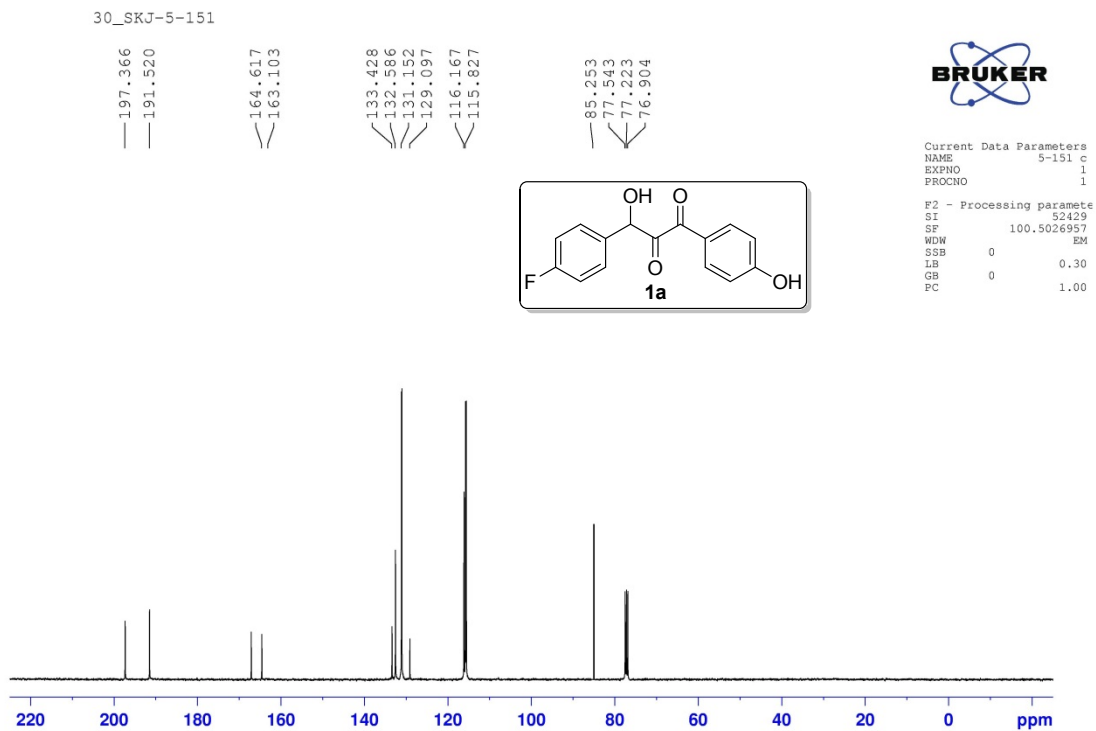
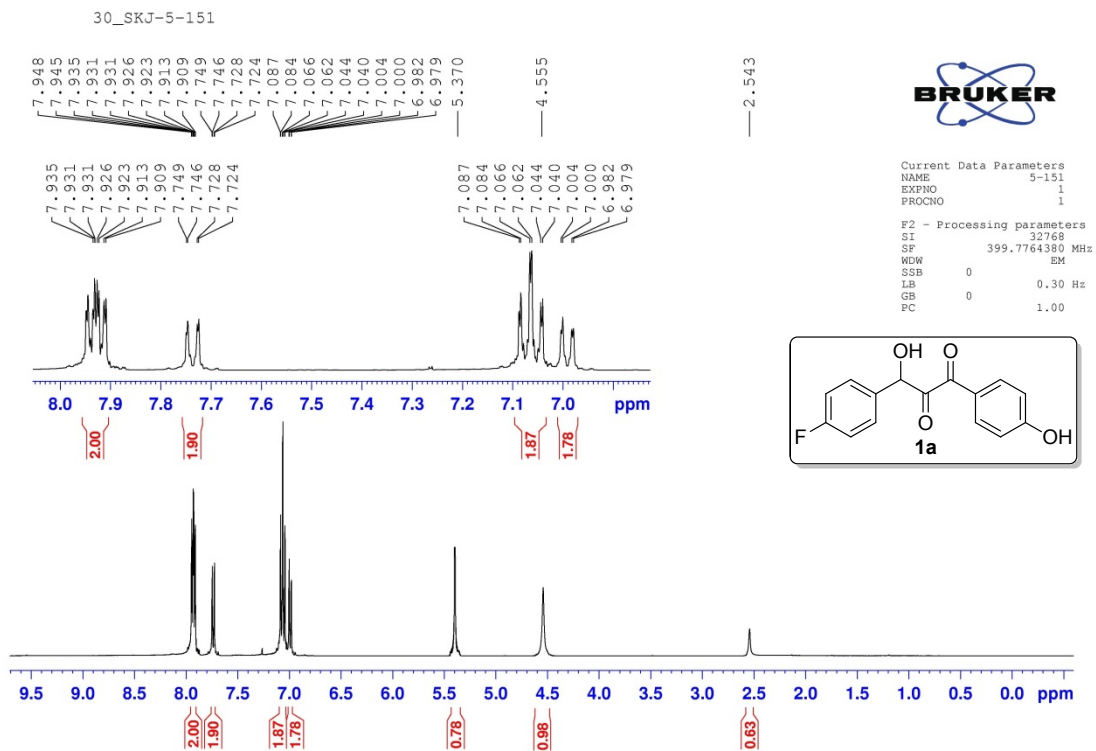
(d, $J = 32$, 11.5 Hz, 2H), 4.07 (tt, $J = 10.5$, 3 Hz, 1H), 2.30-2.35 (m, 2H), 2.20 (s, br, D₂O exchangeable, 1H), 1.77-1.86 (m, 2H). ¹³C NMR (125 MHz, CDCl₃, ppm): δ 189.8, 143.7, 140.7, 135.4, 134.4, 134.1, 129.8, 129.2, 128.5, 128.4, 127.8, 127.5, 126.2, 125.7, 122.0, 77.8, 77.4, 67.2, 39.8. IR (KBr, cm⁻¹): 3454, 2961, 2878, 1651, 1541, 1091, 801. HRMS (ESIMS): for C₂₆H₂₂Br₂NaO₃ (M+Na)⁺ Anal. calcd. 562.9833; found 562.9853.

2-(4-(4-hydroxy-6-phenyltetrahydro-2H-pyran-2-yl)phenyl)-4H-chromen-4-one (36a)

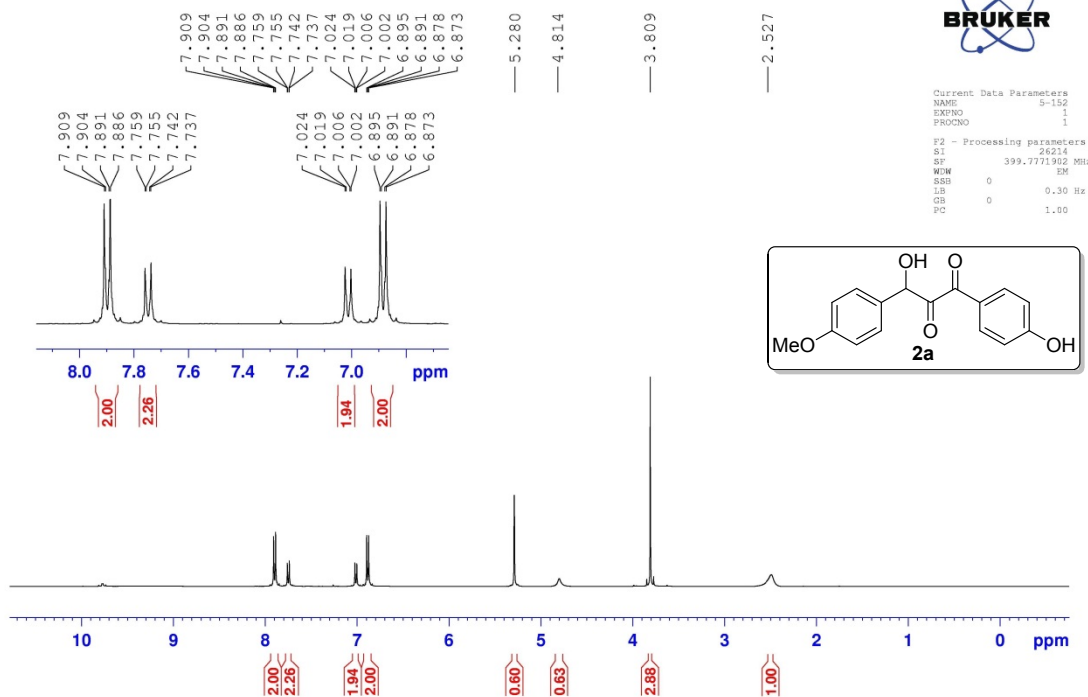


¹H NMR (500 MHz, CDCl₃, ppm): δ 7.74 (d, $J = 8$ Hz, 2H), 7.37 (dd, $J = 6$, 3 Hz, 2H), 7.27 (d, $J = 8.5$ Hz, 2H), 7.17-7.21 (m, 3H), 7.15 (d, $J = 8.5$ Hz, 2H), 6.92 (s, 1H), 6.79 (dd, $J = 6.5$, 3 Hz, 2H), 4.40 (t, $J = 11.5$ Hz, 2H), 3.90 (tt, $J = 11$, 3 Hz, 1H), 2.15-2.22 (m, 2H), 1.68-1.77 (m, 2H). ¹³C NMR (125 MHz, CDCl₃, ppm): δ 190.0, 163.0, 156.0, 139.8, 135.5, 134.1, 131.6, 129.9, 129.3, 128.6, 128.6, 127.6, 127.5, 126.3, 122.2, 121.8, 77.1, 76.9, 65.0, 39.8, 39.7. IR (KBr, cm⁻¹): 3446, 2971, 2880, 1652, 1513, 1208, 799. HRMS (ESIMS): for C₂₆H₂₁NaO₄ (M+Na)⁺ Anal. calcd. 421.1416; found 421.1441.

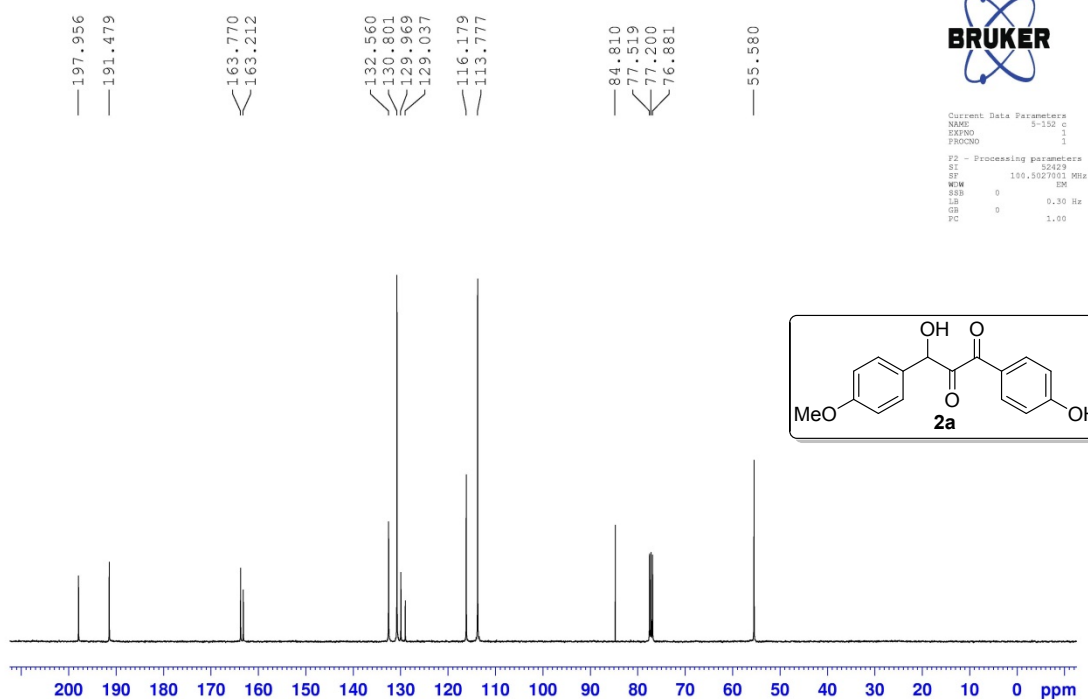
4. ^1H and ^{13}C NMR Spectra of representative compounds.



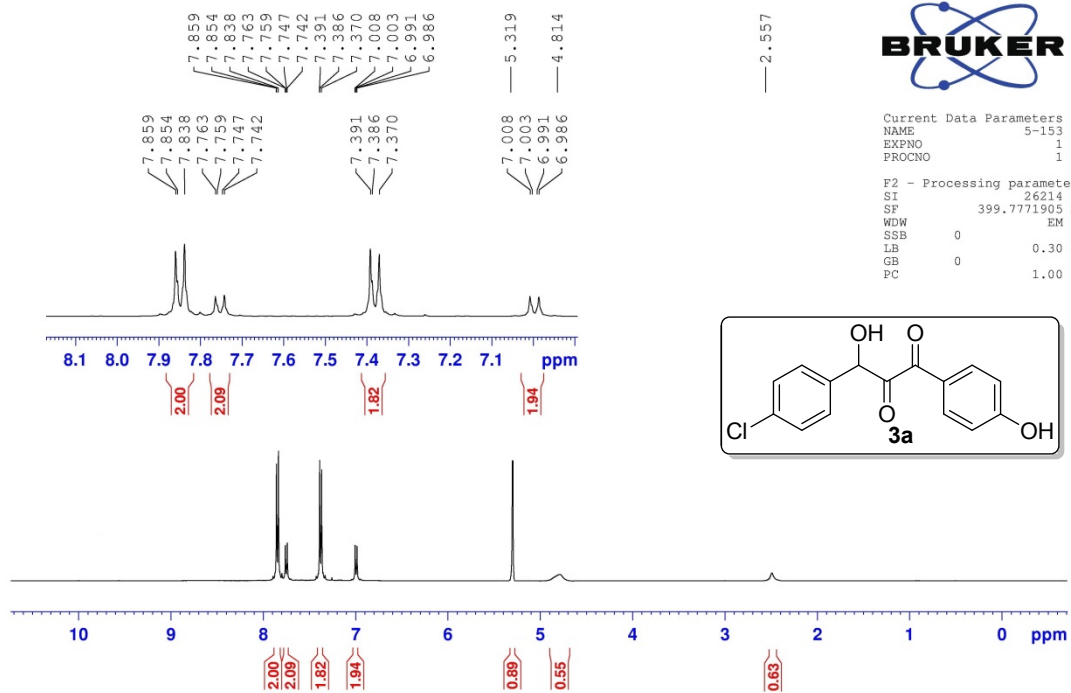
30_SKJ-5-152



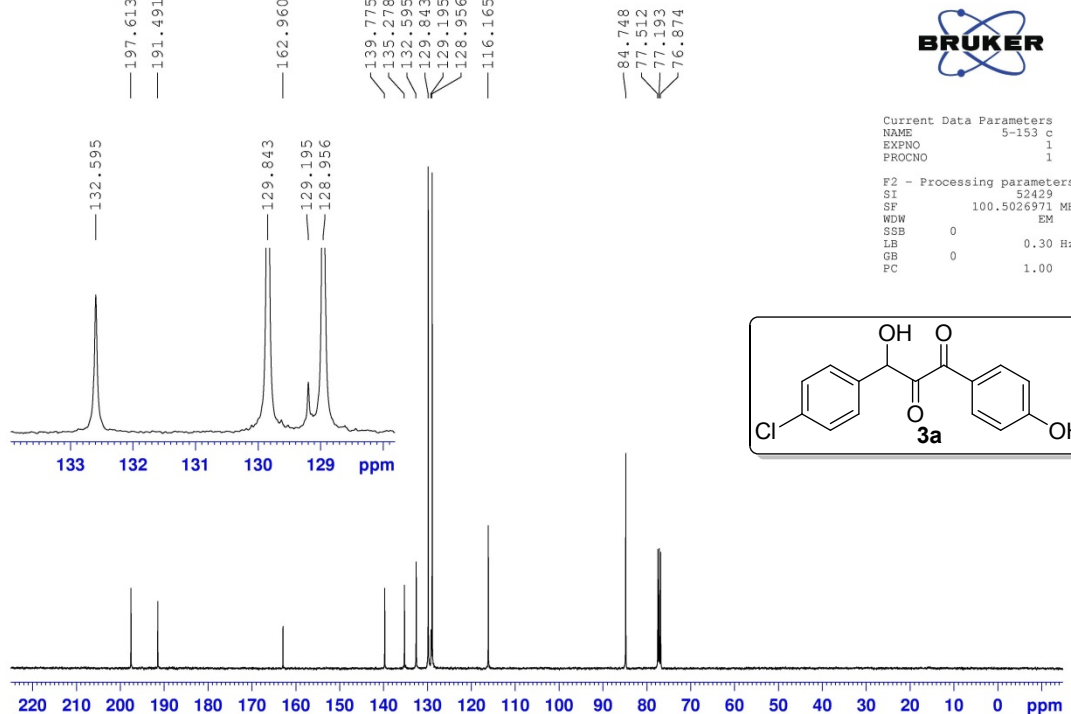
30_SKJ-5-152

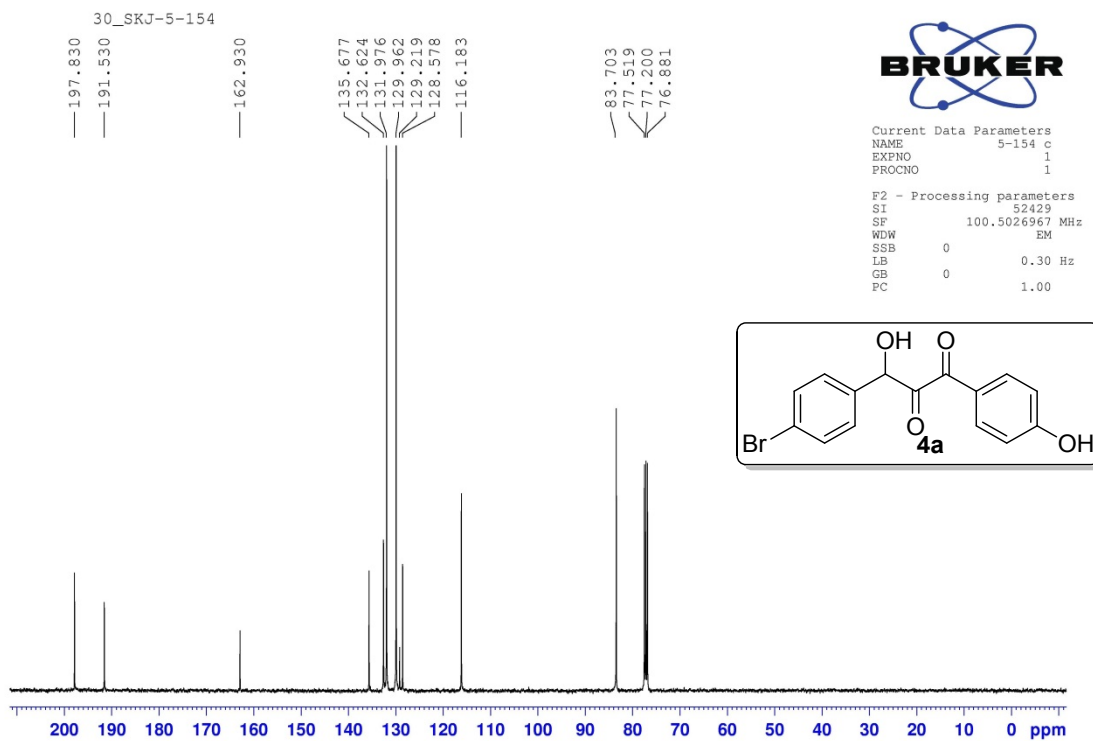
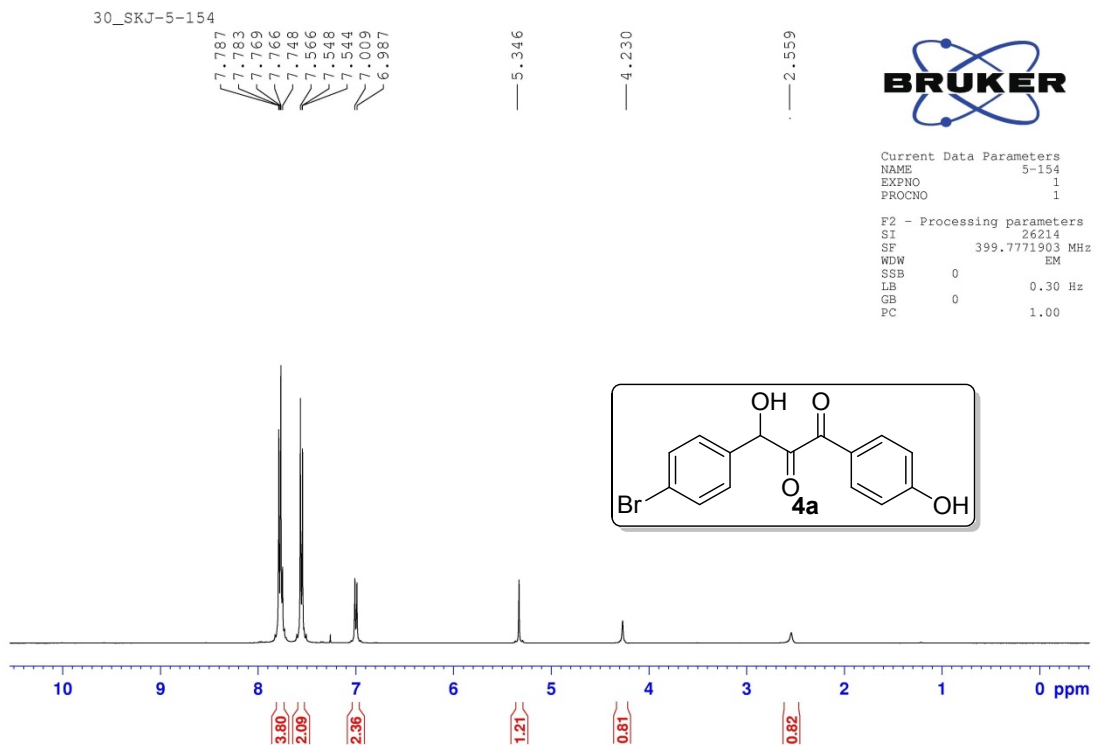


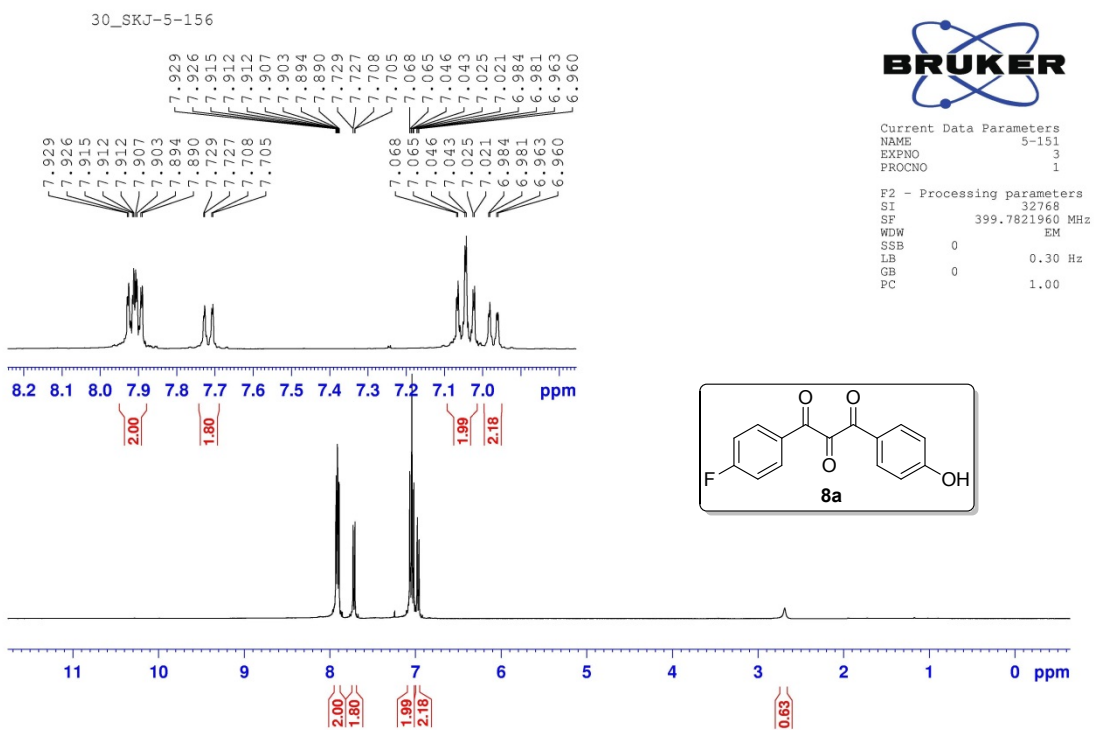
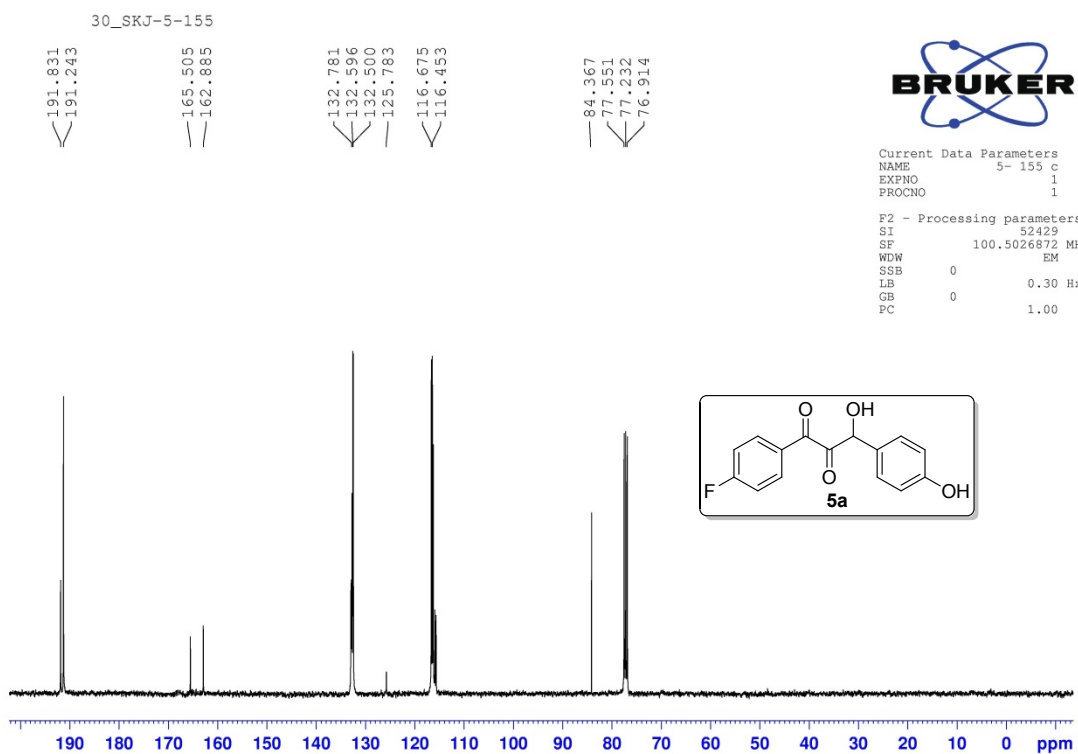
30_SKJ-5-153



30_SKJ-5-153







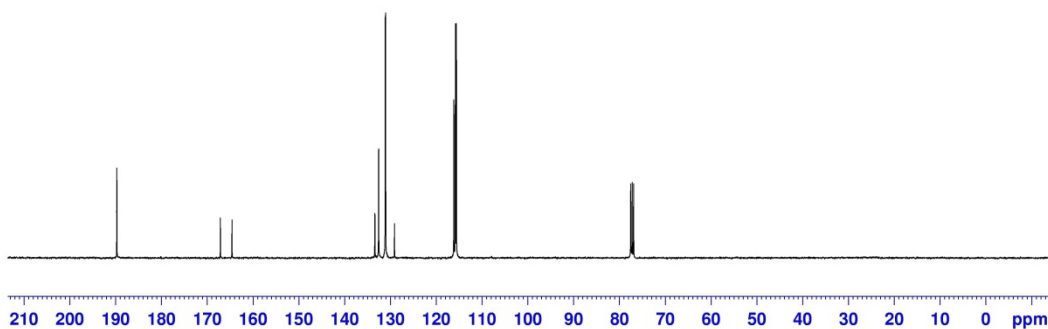
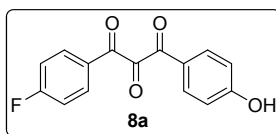
30_SKJ-5-156

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164.748
133.431
132.599
131.061
129.131
116.170
115.830
77.545
77.226
76.907

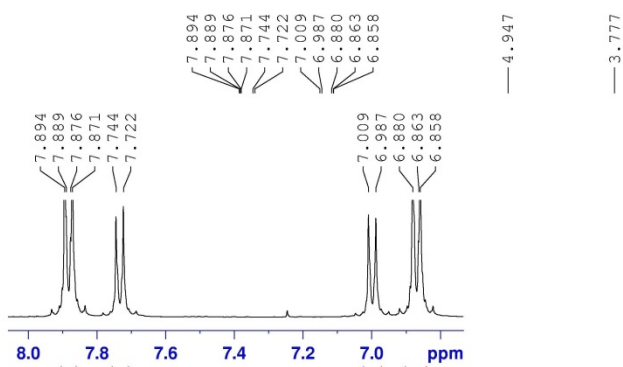


Current Data Parameters
NAME 5-151 c
EXPNO 2
PROCNO 1

F2 - Processing parameters
SI 52429
SF 100.5026954 MHz
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GB 0
PC 1.00

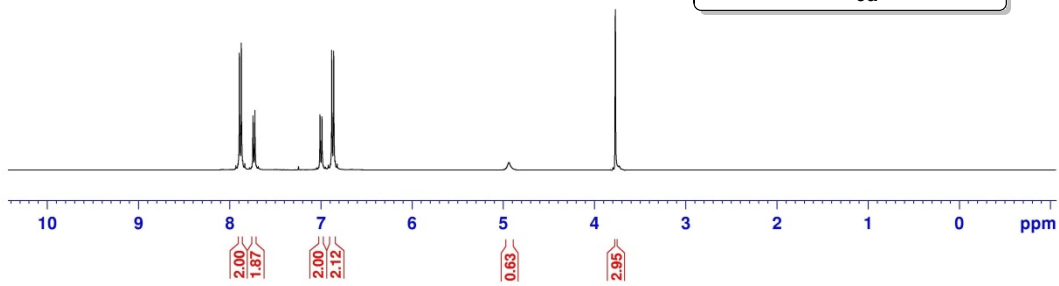
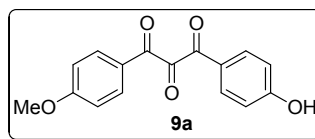


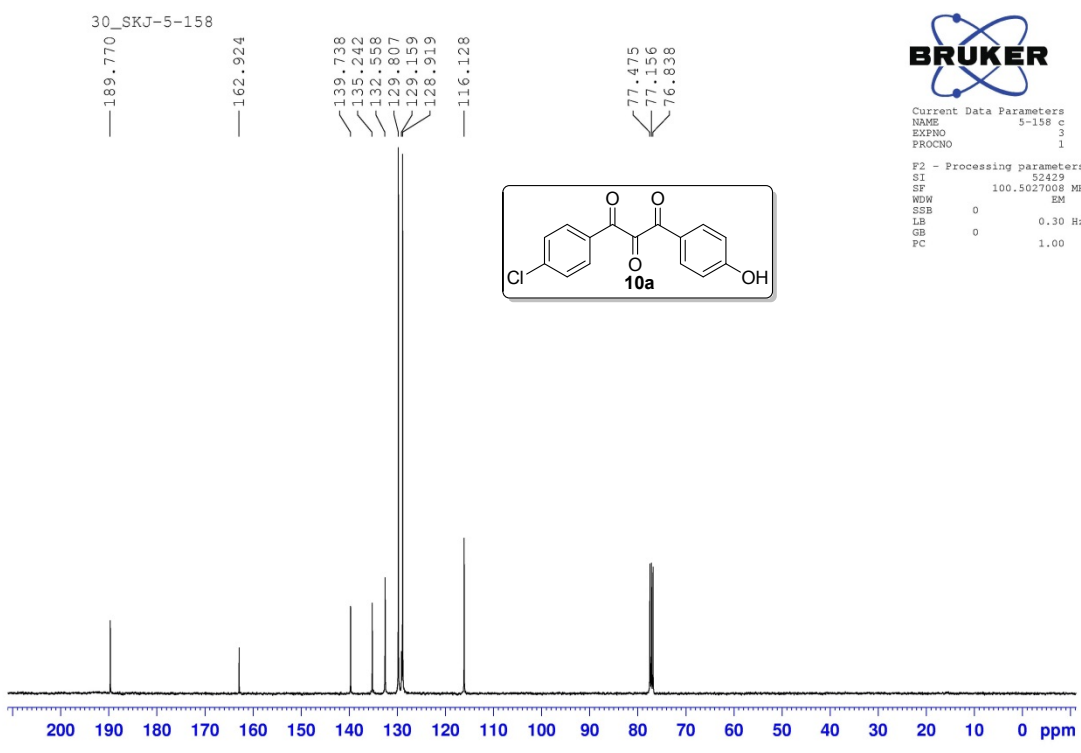
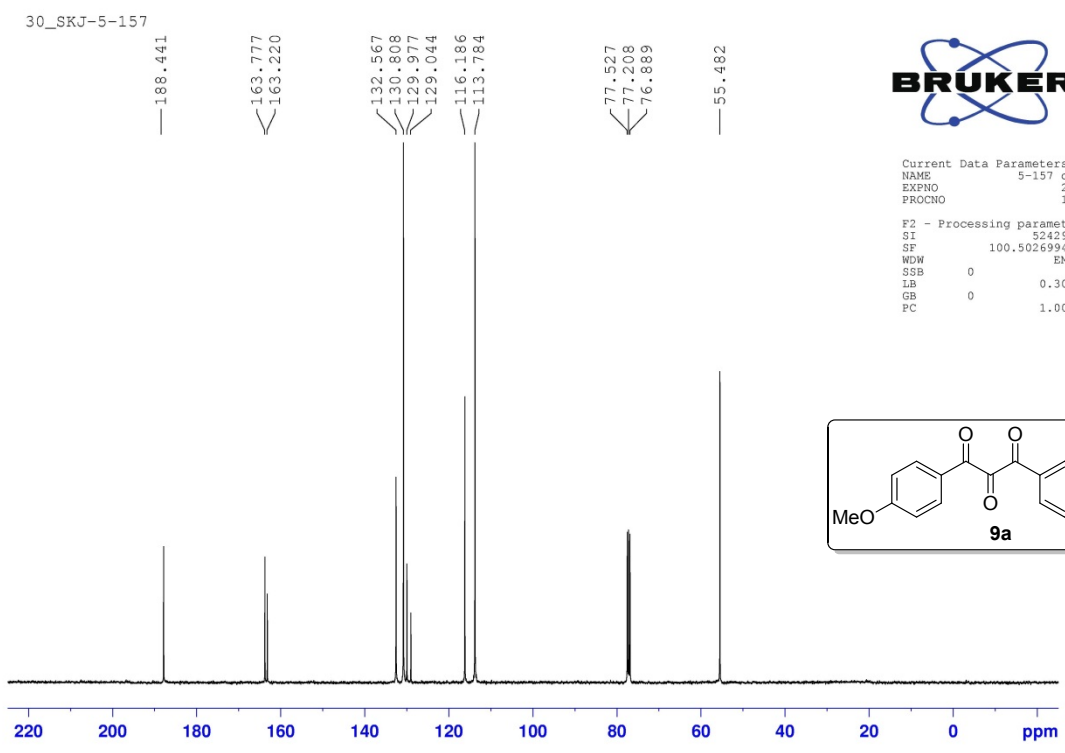
30_SKJ-5-157

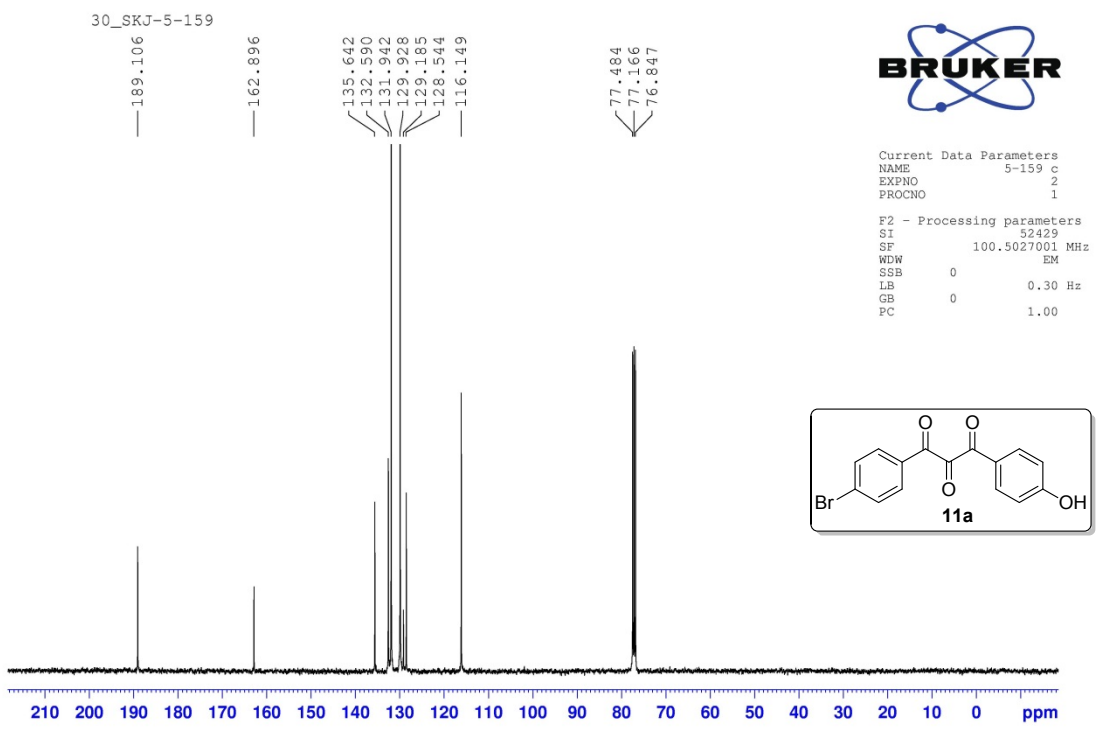
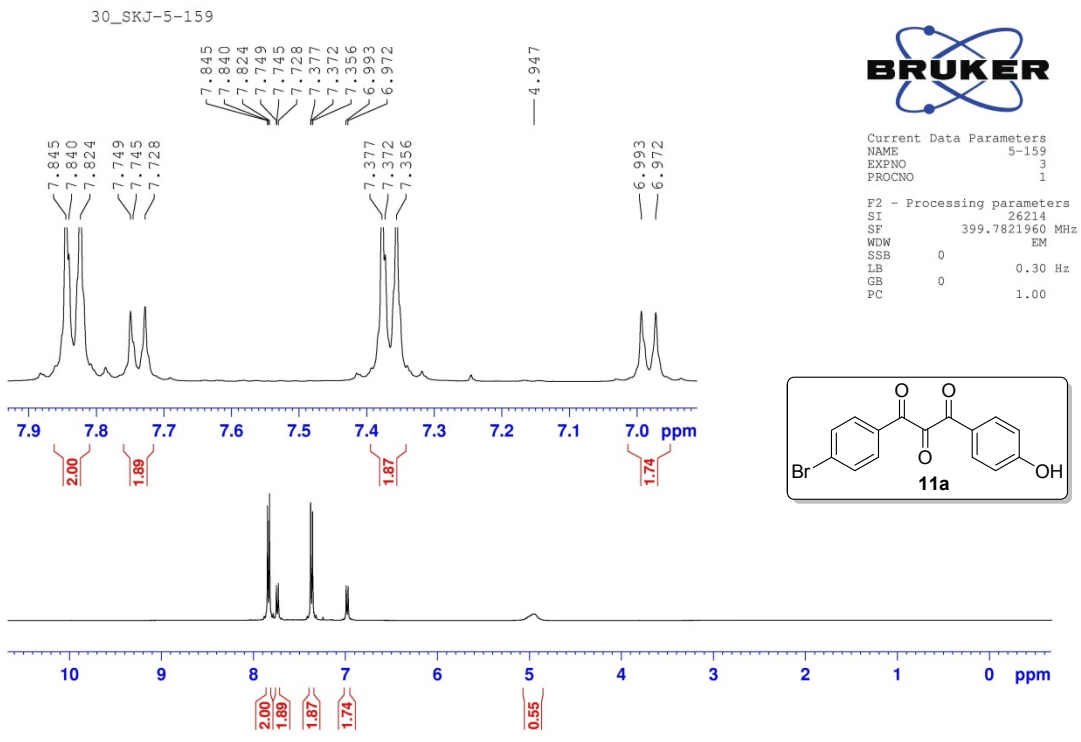


Current Data Parameters
NAME 5-157
EXPNO 2
PROCNO 1

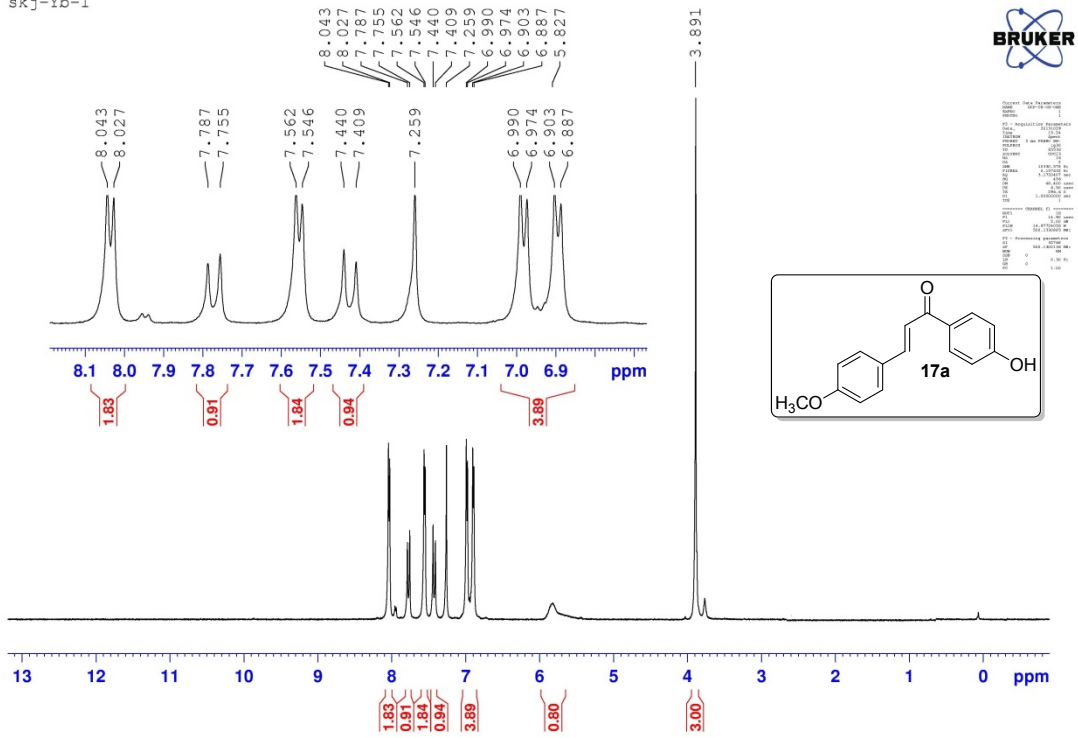
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GB 0
PC 1.00



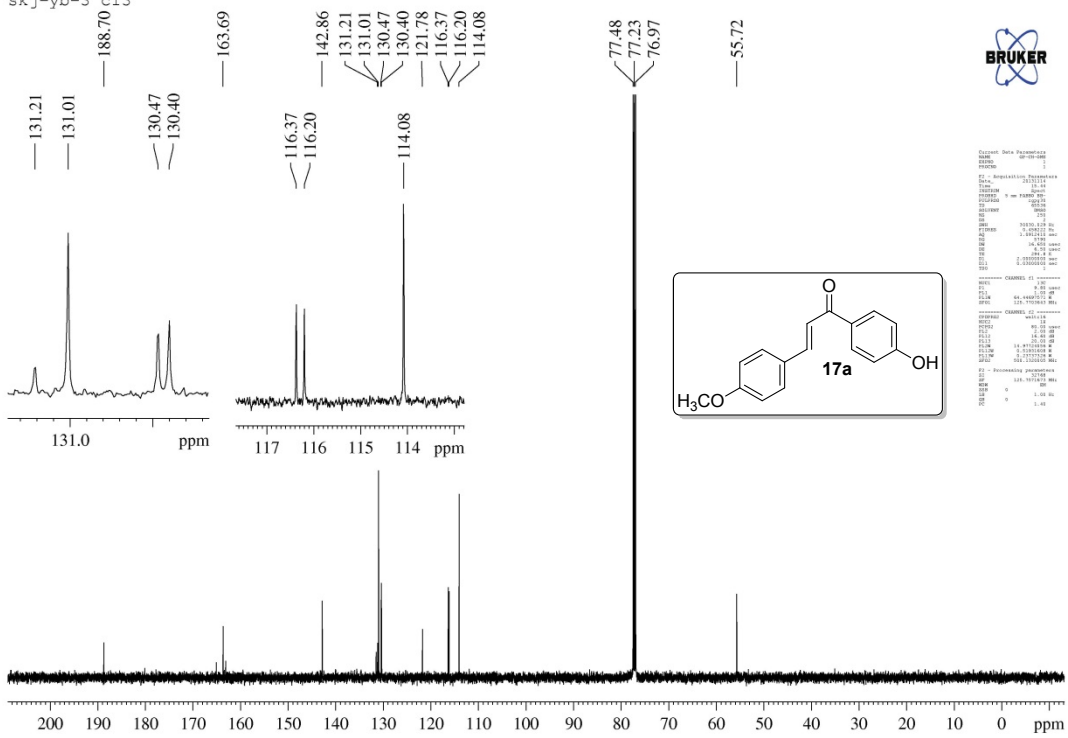


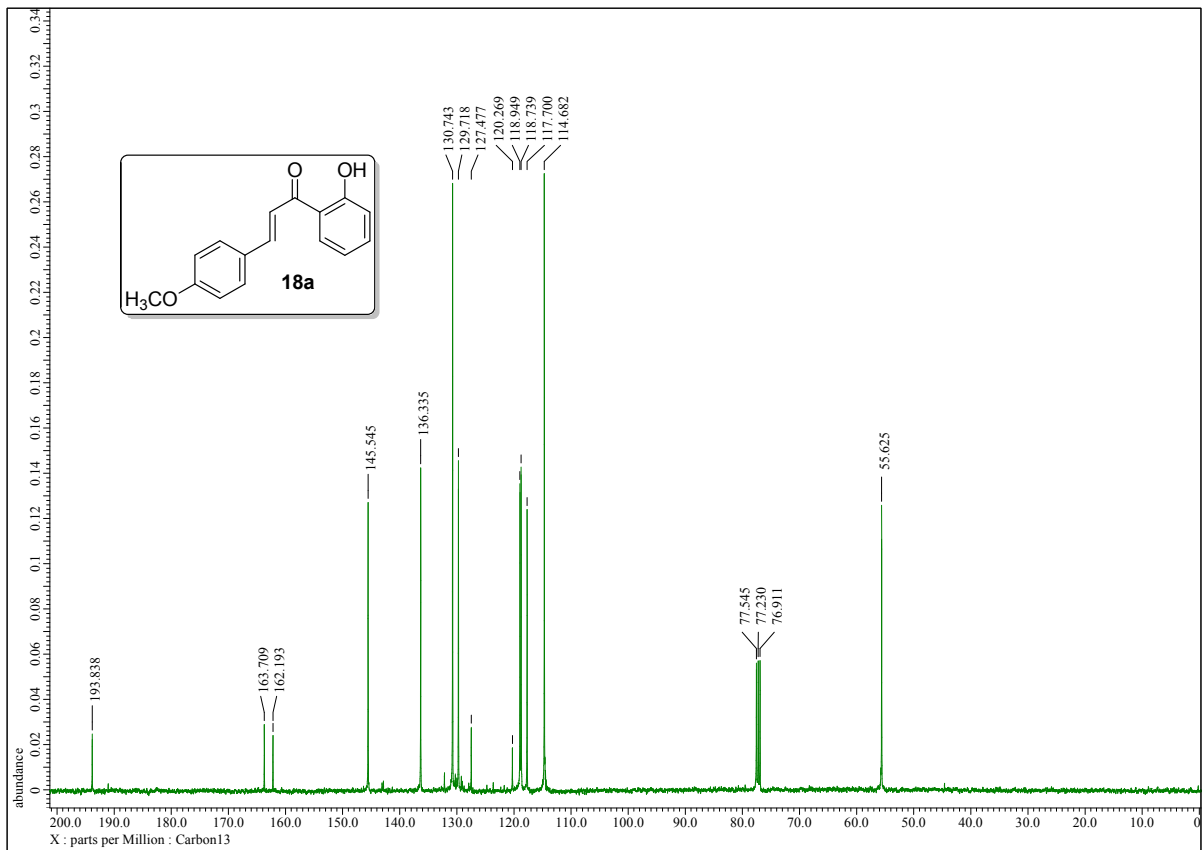
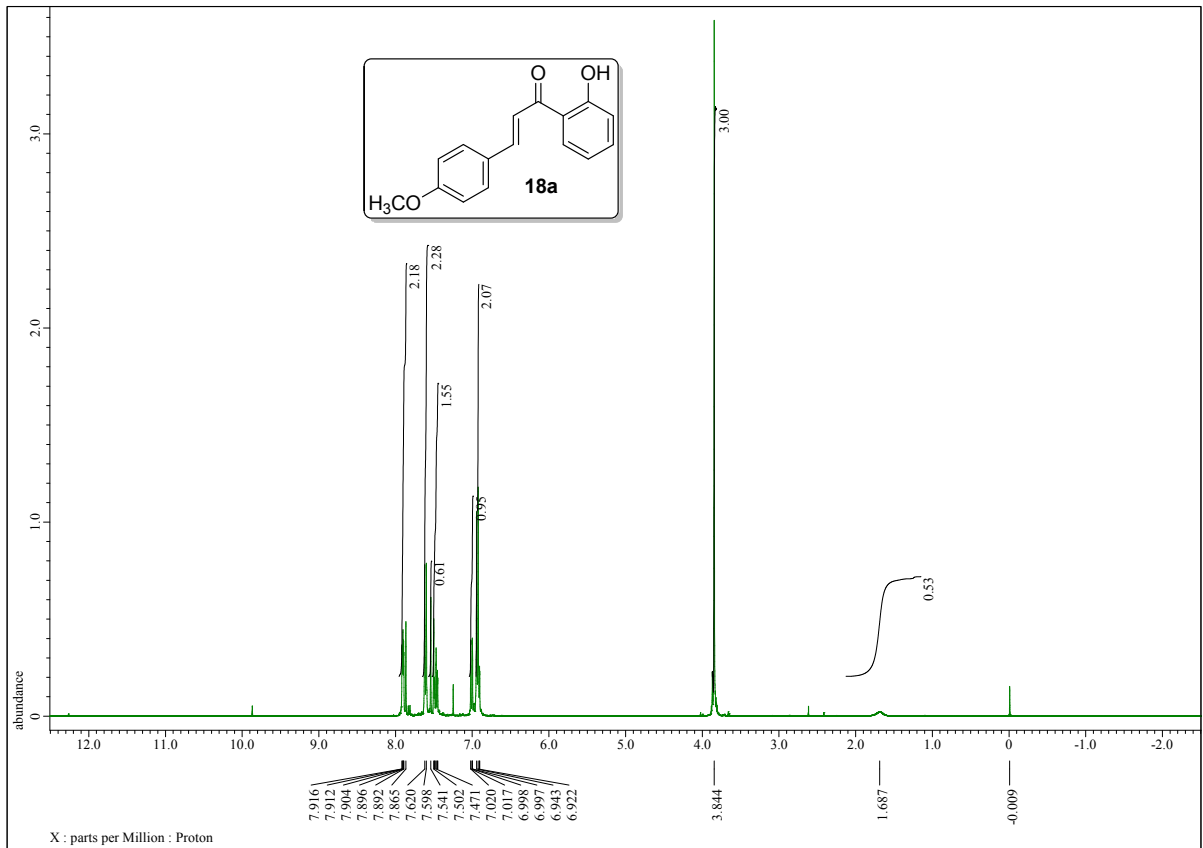


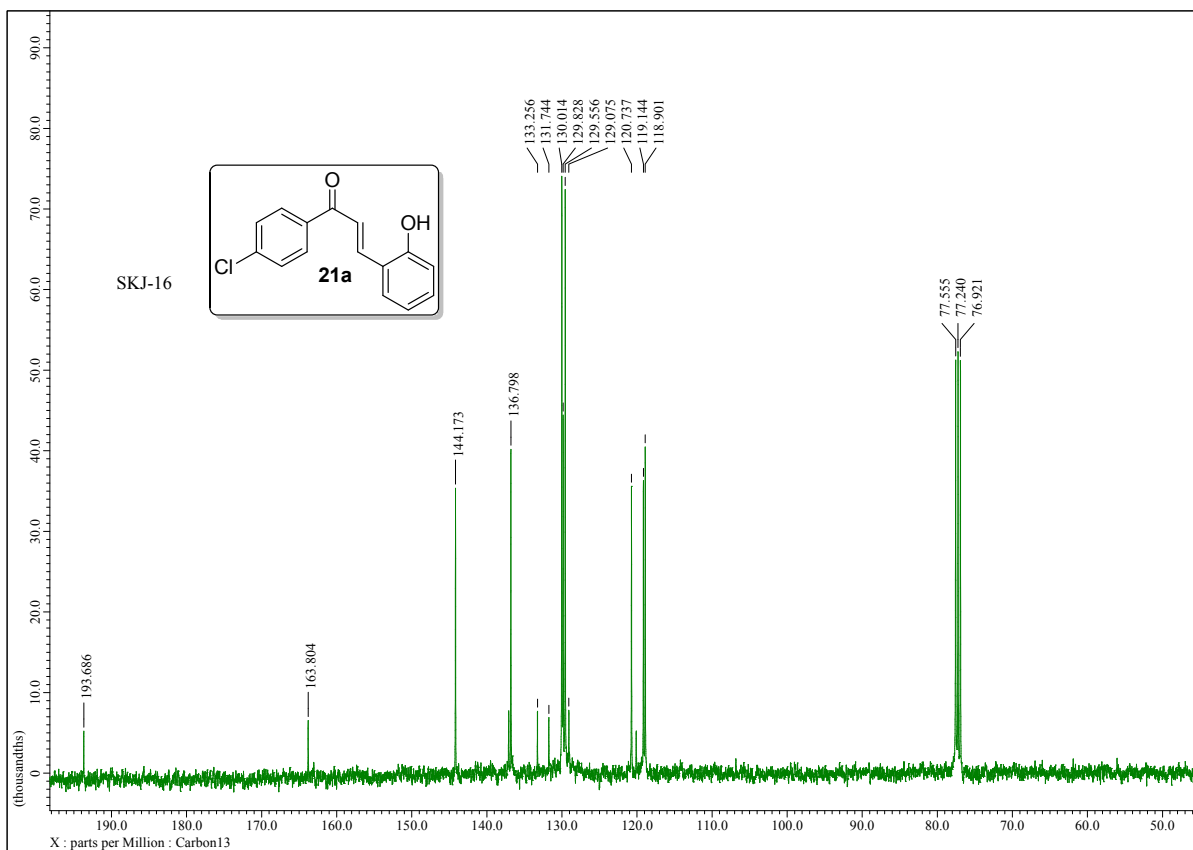
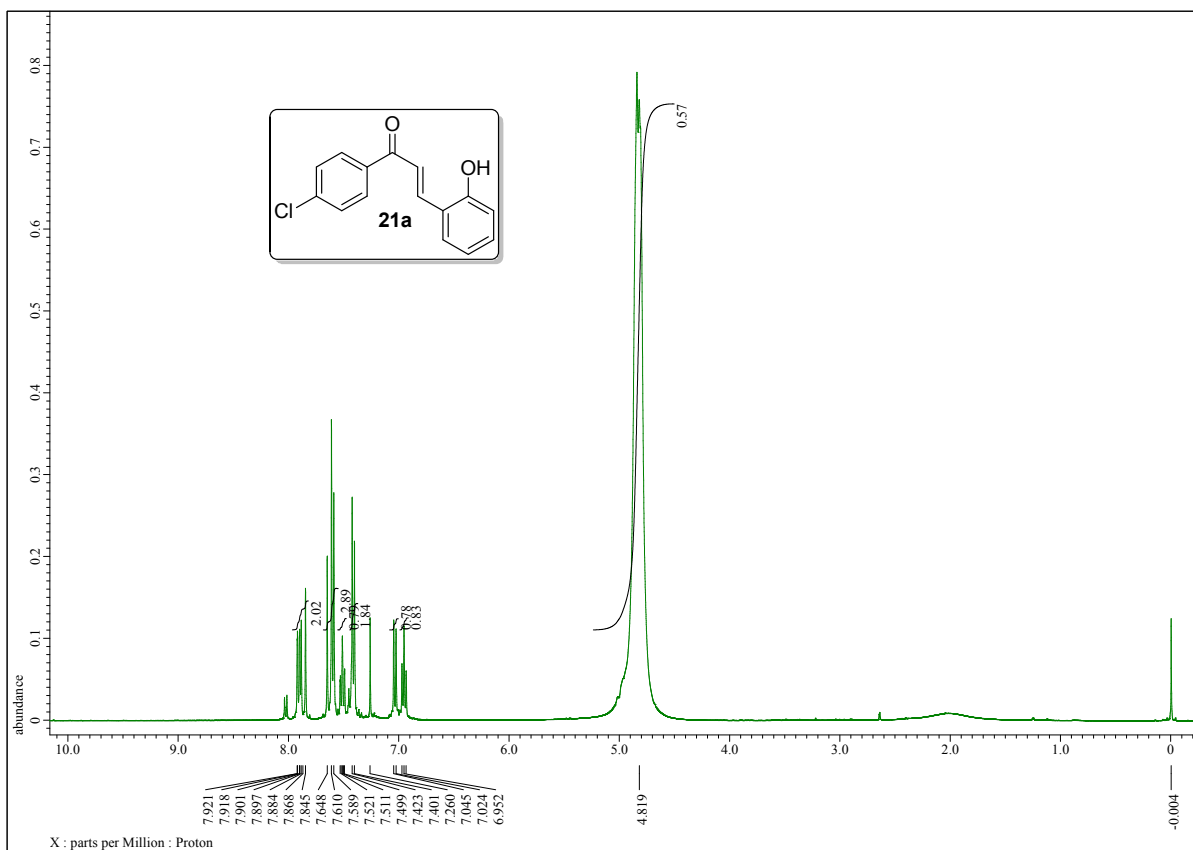
skj-Yb-1



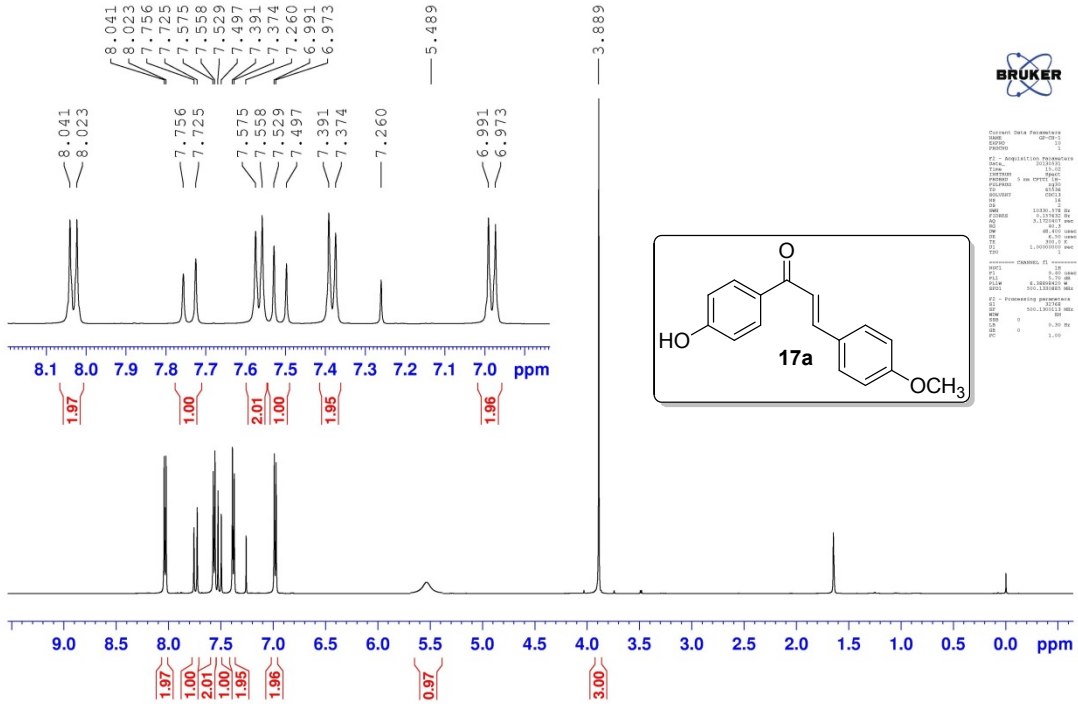
skj-yb-3 c13



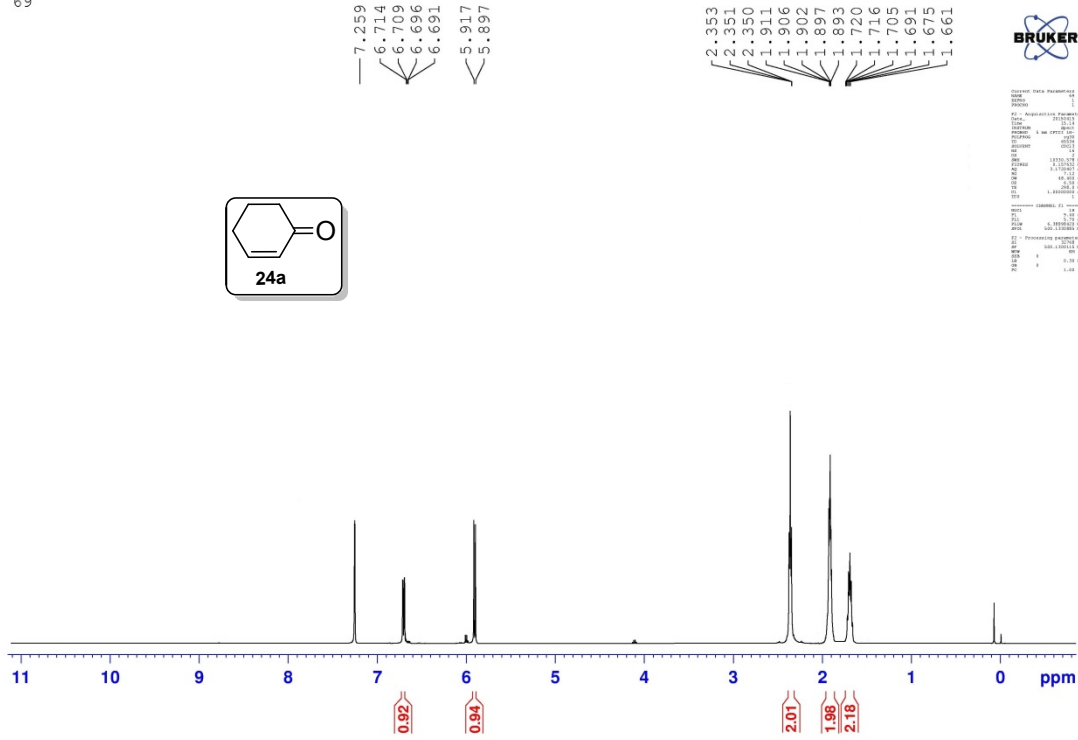




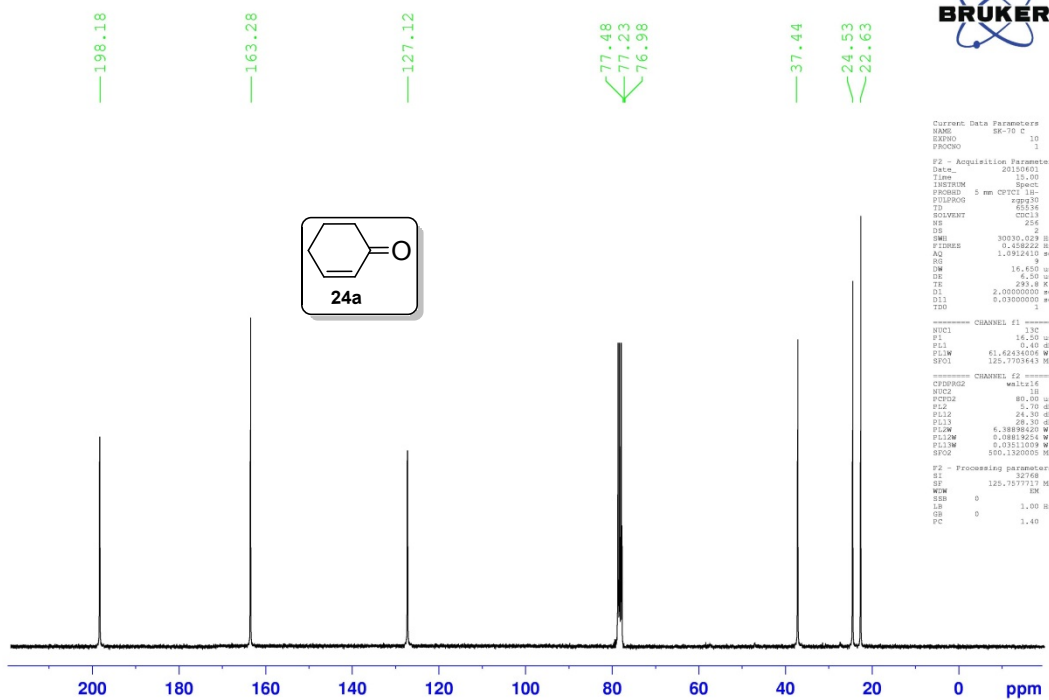
SKJ-YB-3



69

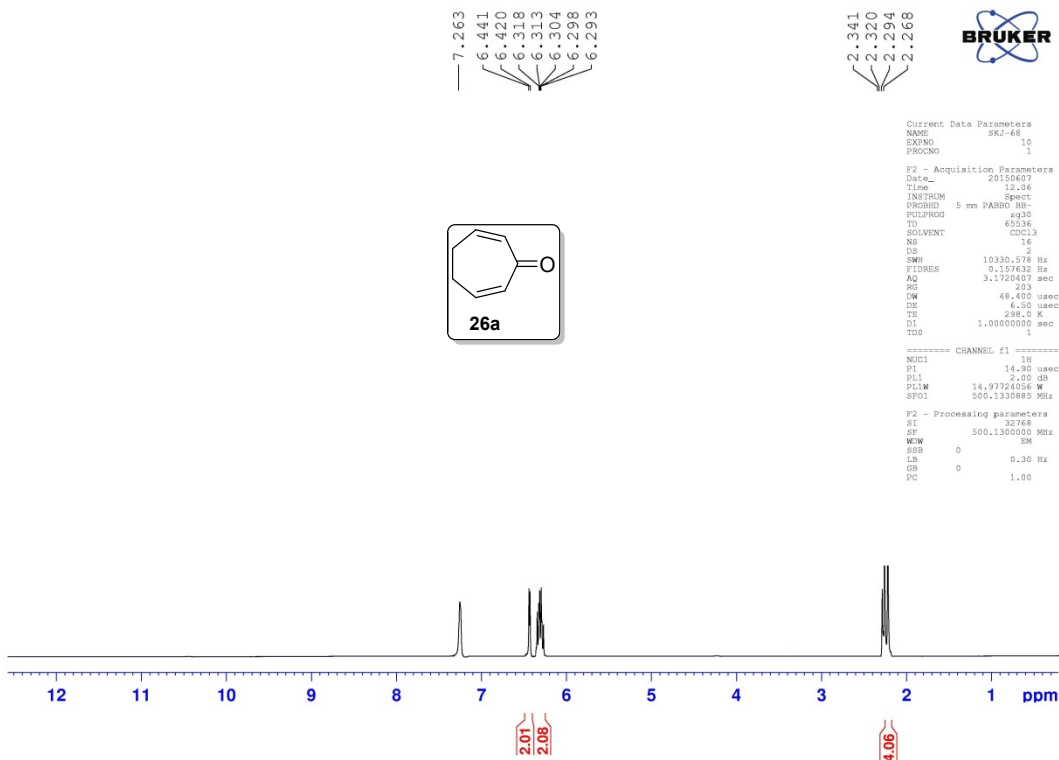


SK-71C



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Current Data Parameters
NAME SKJ-68
EXPNO 10
PROCNO 1
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PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWE 10320.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 203
DW 48.400 usec
DE 6.20 usec
TE 298.0 K
D1 1.0000000 sec
D11 0.0300000 sec
D12 0.0300000 sec
D13 0.0300000 sec
D14 0.0300000 sec
D15 0.0300000 sec
D16 0.0300000 sec
D17 0.0300000 sec
D18 0.0300000 sec
D19 0.0300000 sec
D20 0.0300000 sec
===== CHANNEL f1 =====
NUC1 13C
P1 14.90 usec
PL1 2.00 dB
PL12 14.9724056 W
PL13 14.9724056 W
SFO1 500.1330885 MHz
F2 - Processing parameters
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SF 500.1330885 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
  
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```

Current Data Parameters
NAME SKJ-68
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
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Time 12.06
INSTRUM spect
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PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWE 10320.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 203
DW 48.400 usec
DE 6.20 usec
TE 298.0 K
D1 1.0000000 sec
D11 0.0300000 sec
D12 0.0300000 sec
D13 0.0300000 sec
D14 0.0300000 sec
D15 0.0300000 sec
D16 0.0300000 sec
D17 0.0300000 sec
D18 0.0300000 sec
D19 0.0300000 sec
D20 0.0300000 sec
===== CHANNEL f1 =====
NUC1 1H
P1 14.90 usec
PL1 2.00 dB
PL12 14.9724056 W
PL13 14.9724056 W
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1330885 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
  
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sk-75

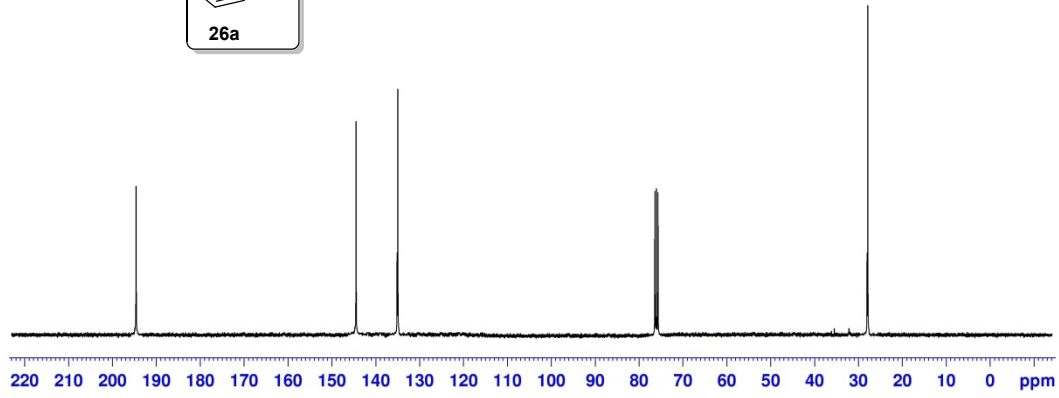
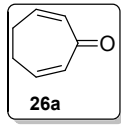
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144.25

135.15

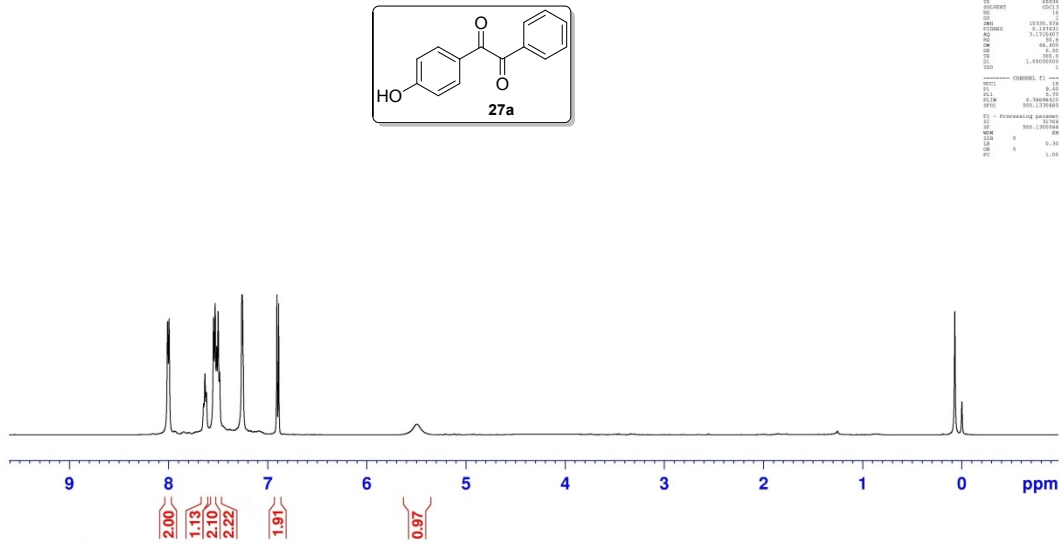
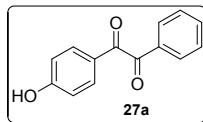
77.48
77.23
76.97

28.13



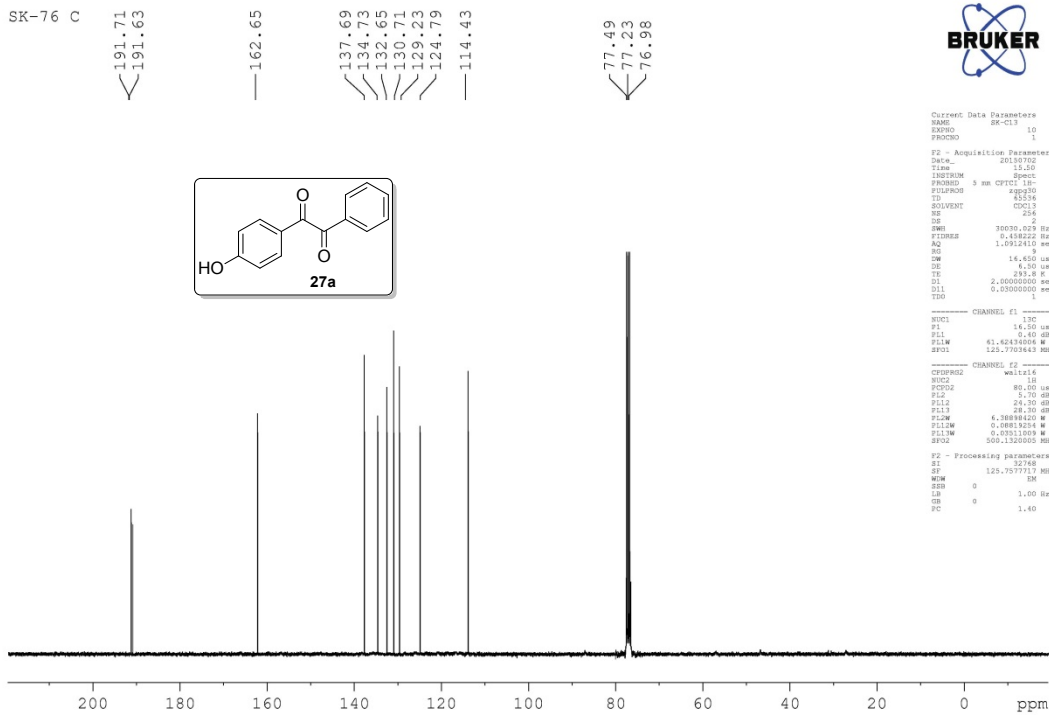
6
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7.648
7.634
7.620
7.548
7.532
7.516
7.501
7.486
7.260
6.991
6.976

5.489



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===== CHANNEL f1 =====
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PL1       0.000000 dB
PL12      0.000000 dB
SFO1      101.253000 MHz
F2 - Processing parameters
SI          32768
SF          500.137000 MHz
AQ          30
SFO         0.000000 MHz
AQ         0.000000 MHz
PC          1.00
```

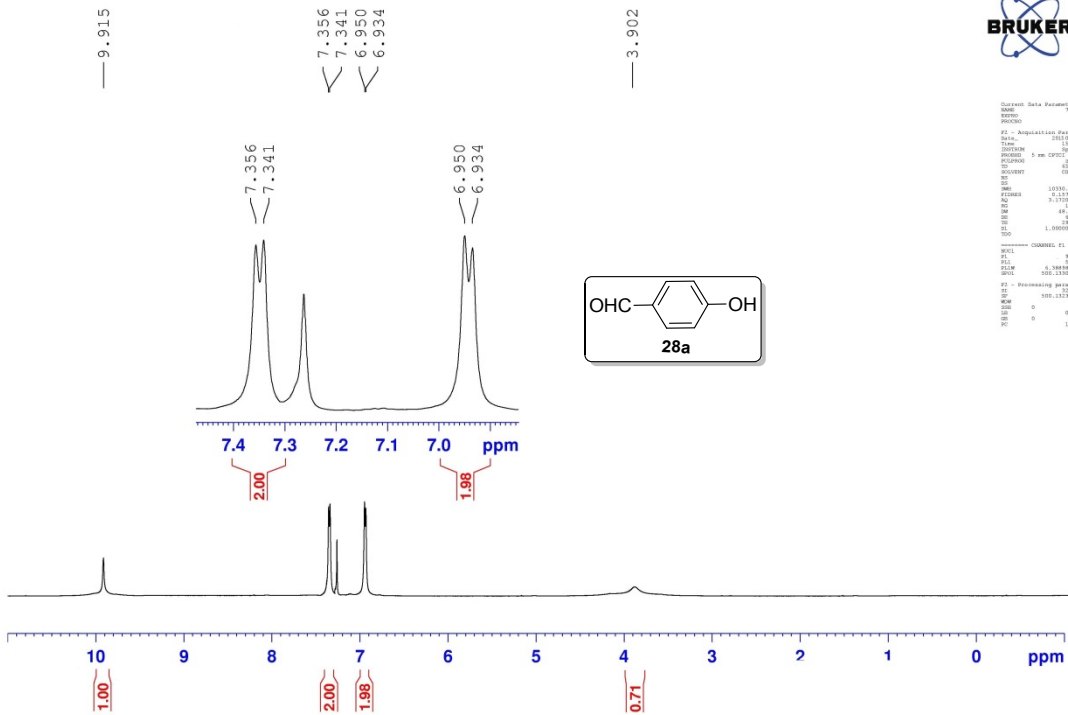
SK-76 C



```

Current Data Parameters
NAME      SK-cl3
EXPNO    10
PROCNO   1
F2 - Acquisition Parameters
Date_    20100702
Time     12.50
INSTRUM  spect
PROBHD   5 mm CPY1 1H-
PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
DE        2.54
DS        2
SFO      300.135 MHz
FIDRES   0.189222 Hz
AQ        1.0722410 sec
RG         3
WDW        16.450 usec
DE         6.50 usec
TE        293.2 K
D1         2.0000000 sec
D11        0.0300000 sec
D10        1
----- CHANNEL f1 -----
NUC1      13C
P1         14.50 usec
PL1        0.40 dB
SFO1     125.7703443 MHz
----- CHANNEL f2 -----
CFPRPG   waltz16
NUC2      1H
PCPDZ    80.00 usec
PL2       5.70 dB
PL12      2.30 dB
PL13      2.30 dB
PL14      2.30 dB
PL15      2.30 dB
PL16      2.30 dB
PL17      2.30 dB
PL18      2.30 dB
PL19      2.30 dB
PL20      2.30 dB
PL21      2.30 dB
PL22      2.30 dB
PL23      2.30 dB
PL24      2.30 dB
PL25      2.30 dB
PL26      2.30 dB
PL27      2.30 dB
PL28      2.30 dB
PL29      2.30 dB
PL30      2.30 dB
PL31      2.30 dB
PL32      2.30 dB
PL33      2.30 dB
PL34      2.30 dB
PL35      2.30 dB
PL36      2.30 dB
PL37      2.30 dB
PL38      2.30 dB
PL39      2.30 dB
PL40      2.30 dB
PL41      2.30 dB
PL42      2.30 dB
PL43      2.30 dB
PL44      2.30 dB
PL45      2.30 dB
PL46      2.30 dB
PL47      2.30 dB
PL48      2.30 dB
PL49      2.30 dB
PL50      2.30 dB
PL51      2.30 dB
PL52      2.30 dB
PL53      2.30 dB
PL54      2.30 dB
PL55      2.30 dB
PL56      2.30 dB
PL57      2.30 dB
PL58      2.30 dB
PL59      2.30 dB
PL60      2.30 dB
PL61      2.30 dB
PL62      2.30 dB
PL63      2.30 dB
PL64      2.30 dB
PL65      2.30 dB
PL66      2.30 dB
PL67      2.30 dB
PL68      2.30 dB
PL69      2.30 dB
PL70      2.30 dB
PL71      2.30 dB
PL72      2.30 dB
PL73      2.30 dB
PL74      2.30 dB
PL75      2.30 dB
PL76      2.30 dB
PL77      2.30 dB
PL78      2.30 dB
PL79      2.30 dB
PL80      2.30 dB
PL81      2.30 dB
PL82      2.30 dB
PL83      2.30 dB
PL84      2.30 dB
PL85      2.30 dB
PL86      2.30 dB
PL87      2.30 dB
PL88      2.30 dB
PL89      2.30 dB
PL90      2.30 dB
PL91      2.30 dB
PL92      2.30 dB
PL93      2.30 dB
PL94      2.30 dB
PL95      2.30 dB
PL96      2.30 dB
PL97      2.30 dB
PL98      2.30 dB
PL99      2.30 dB
PL100     2.30 dB
----- Processing parameters -----
SI         32768
SF         125.767717 MHz
WDW        EM
SSB         0
LB          1.00 Hz
GB          0
PC          1.40
  
```

74-2

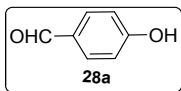


```

Current Data Parameters
NAME      74-2
EXPNO    1
PROCNO   1
F2 - Acquisition Parameters
Date_    20100702
Time     12.50
INSTRUM  spect
PROBHD   5 mm CPY1 1H-
PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
DE        2.54
DS        2
SFO      300.135 MHz
FIDRES   0.189222 Hz
AQ        1.0722410 sec
RG         3
WDW        16.450 usec
DE         6.50 usec
TE        293.2 K
D1         2.0000000 sec
D11        0.0300000 sec
D10        1
----- CHANNEL f1 -----
NUC1      1H
P1         14.50 usec
PL1        0.40 dB
SFO1     125.7703443 MHz
----- CHANNEL f2 -----
CFPRPG   waltz16
NUC2      13C
PCPDZ    80.00 usec
PL2       5.70 dB
PL12      2.30 dB
PL13      2.30 dB
PL14      2.30 dB
PL15      2.30 dB
PL16      2.30 dB
PL17      2.30 dB
PL18      2.30 dB
PL19      2.30 dB
PL20      2.30 dB
PL21      2.30 dB
PL22      2.30 dB
PL23      2.30 dB
PL24      2.30 dB
PL25      2.30 dB
PL26      2.30 dB
PL27      2.30 dB
PL28      2.30 dB
PL29      2.30 dB
PL30      2.30 dB
PL31      2.30 dB
PL32      2.30 dB
PL33      2.30 dB
PL34      2.30 dB
PL35      2.30 dB
PL36      2.30 dB
PL37      2.30 dB
PL38      2.30 dB
PL39      2.30 dB
PL40      2.30 dB
PL41      2.30 dB
PL42      2.30 dB
PL43      2.30 dB
PL44      2.30 dB
PL45      2.30 dB
PL46      2.30 dB
PL47      2.30 dB
PL48      2.30 dB
PL49      2.30 dB
PL50      2.30 dB
PL51      2.30 dB
PL52      2.30 dB
PL53      2.30 dB
PL54      2.30 dB
PL55      2.30 dB
PL56      2.30 dB
PL57      2.30 dB
PL58      2.30 dB
PL59      2.30 dB
PL60      2.30 dB
PL61      2.30 dB
PL62      2.30 dB
PL63      2.30 dB
PL64      2.30 dB
PL65      2.30 dB
PL66      2.30 dB
PL67      2.30 dB
PL68      2.30 dB
PL69      2.30 dB
PL70      2.30 dB
PL71      2.30 dB
PL72      2.30 dB
PL73      2.30 dB
PL74      2.30 dB
PL75      2.30 dB
PL76      2.30 dB
PL77      2.30 dB
PL78      2.30 dB
PL79      2.30 dB
PL80      2.30 dB
PL81      2.30 dB
PL82      2.30 dB
PL83      2.30 dB
PL84      2.30 dB
PL85      2.30 dB
PL86      2.30 dB
PL87      2.30 dB
PL88      2.30 dB
PL89      2.30 dB
PL90      2.30 dB
PL91      2.30 dB
PL92      2.30 dB
PL93      2.30 dB
PL94      2.30 dB
PL95      2.30 dB
PL96      2.30 dB
PL97      2.30 dB
PL98      2.30 dB
PL99      2.30 dB
PL100     2.30 dB
----- Processing parameters -----
SI         32768
SF         125.767717 MHz
WDW        EM
SSB         0
LB          1.00 Hz
GB          0
PC          1.40
  
```

sk-75 c

— 192.90
 — 163.07
 — 134.73
 — 130.29
 — 117.18
 < 77.48
 < 77.23
 < 76.97



```

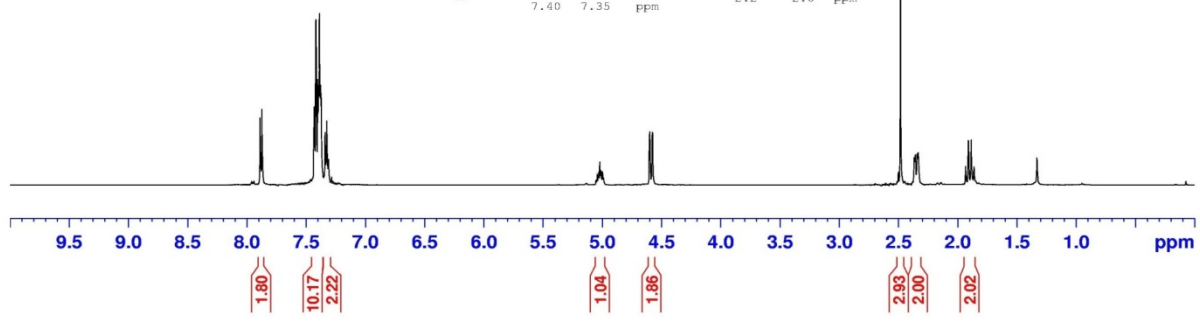
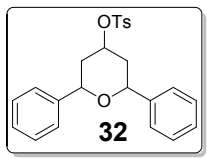
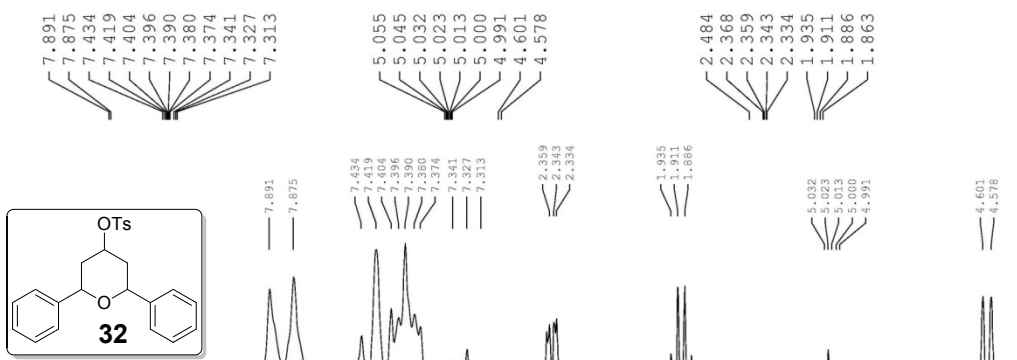
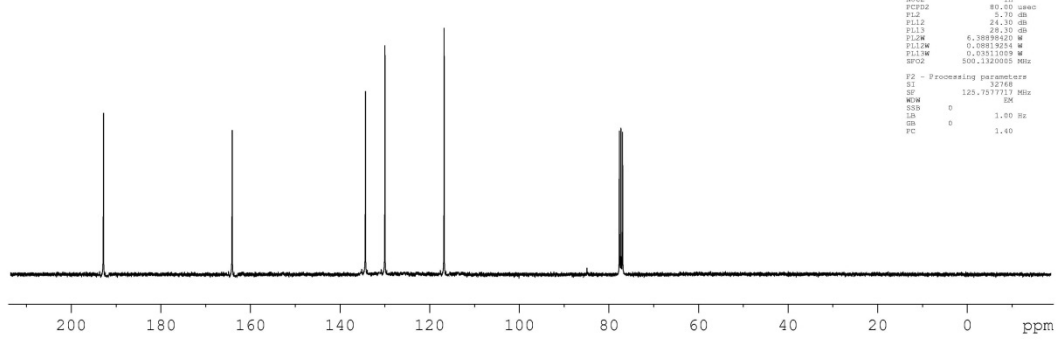
Current Data Parameters
NAME          C13
EXPNO        10
PROCNO       1

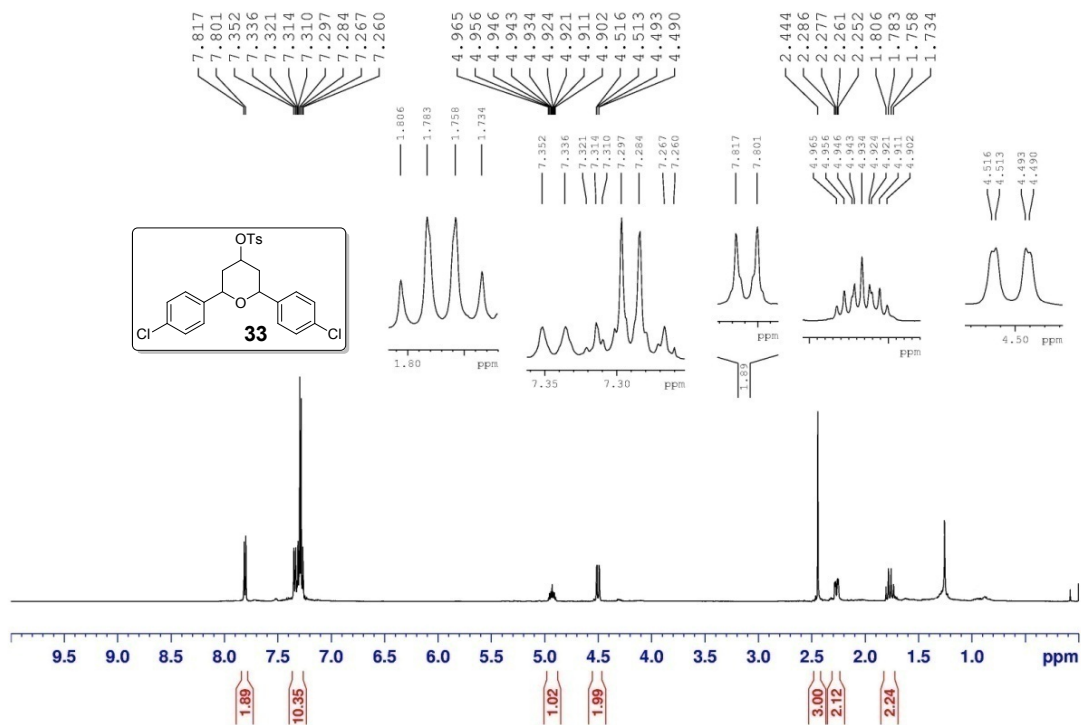
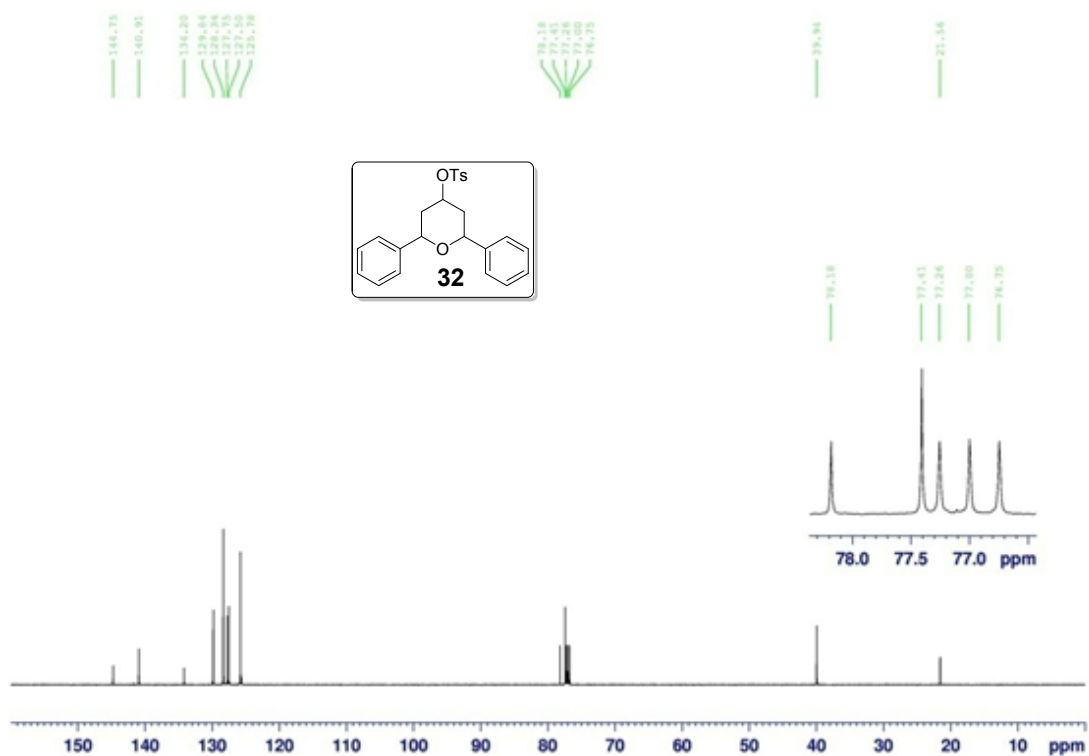
F2 - Acquisition Parameters
Date_        20100702
Time         19.10
INSTRUM      spect
PROBHD       5 mm CPYCI 13C
PULPROG      zgpg30
TD           65536
SOLVENT      CDCl3
NS           256
DS           4
SFO1         30013.029 Hz
FIDRES       0.449222 Hz
AQ           1.0092410 sec
RG           3
AQ           16.450 usec
DE           4.10 usec
TE           293.2 K
D1           2.00000000 sec
d11          0.00000000 sec
TD0          1

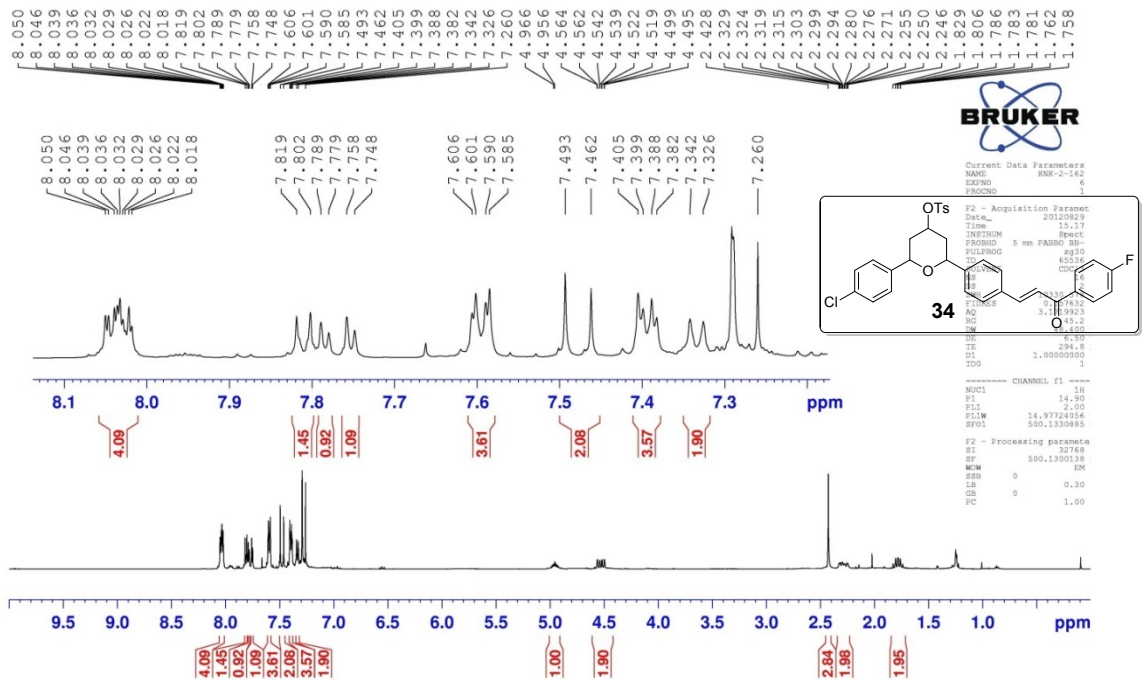
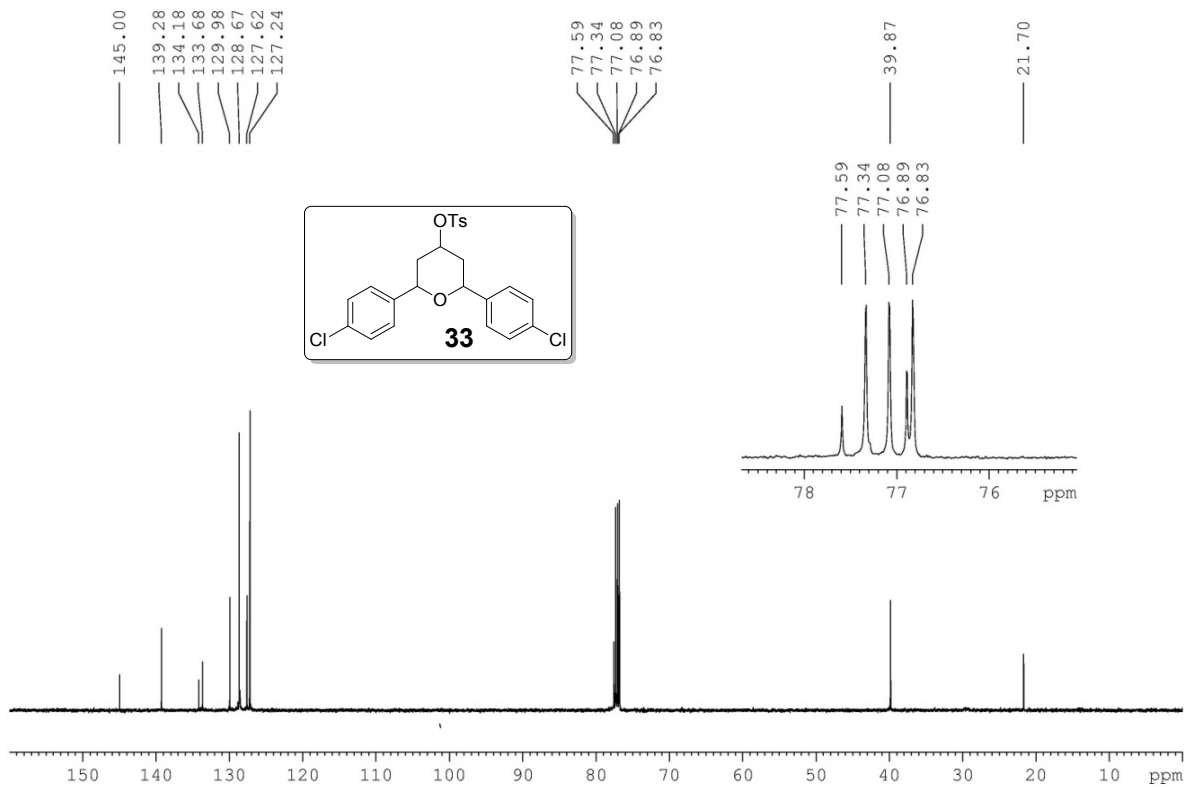
----- CHANNEL f1 -----
NUC1         13C
P1           16.00 usec
PL1         0.40 dB
PL12        63.6241406 Hz
SFO1        125.7703643 MHz

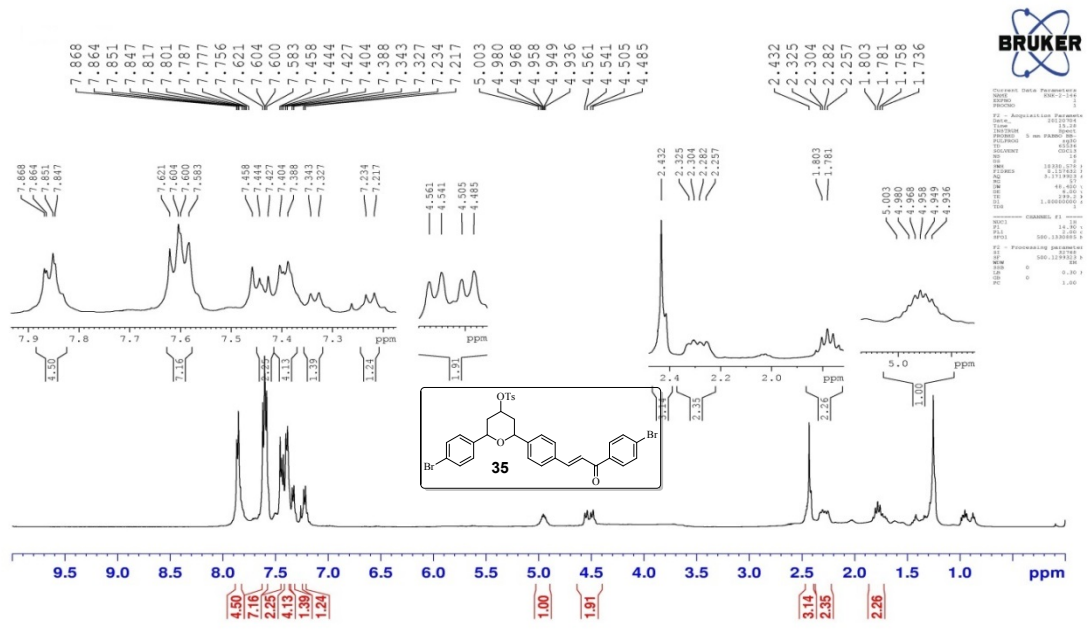
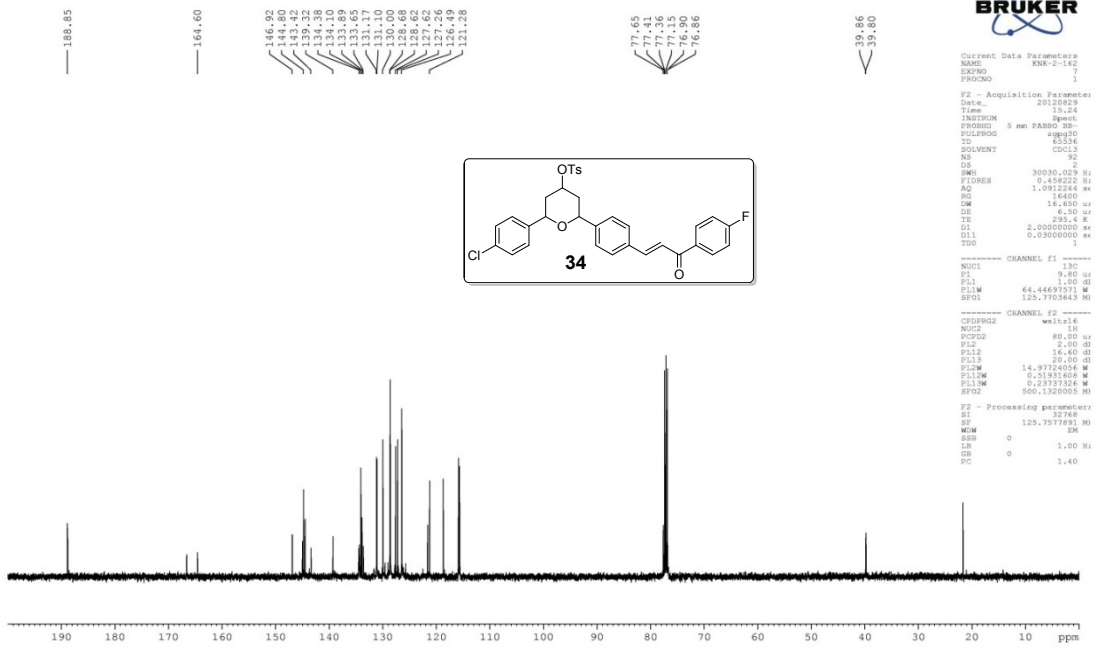
----- CHANNEL f2 -----
CPDPRG2     waltz16
NUC2         1H
PCPDZ       80.00 usec
PL2         2.70 dB
PL12        24.30 dB
PL13        28.30 dB
PL14        6.3889620 Hz
PL15        0.8819254 Hz
PL16        0.0201209 Hz
SFO2        500.1320085 MHz

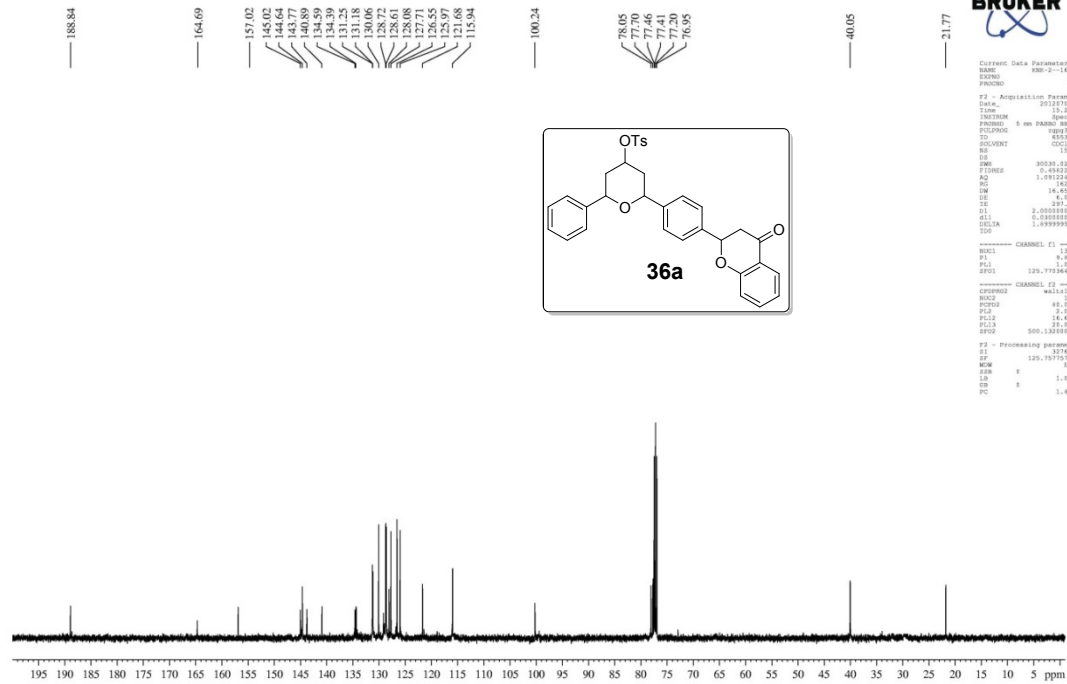
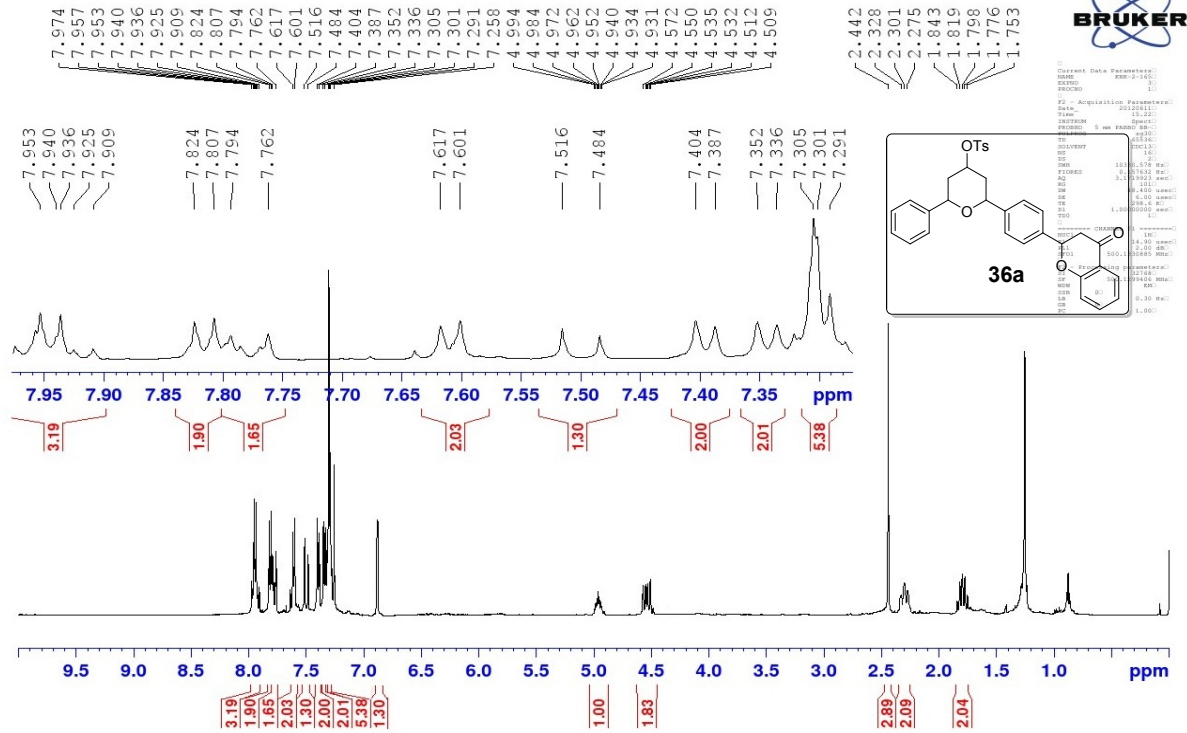
F2 - Processing parameters
SI           32768
SF           125.7577117 MHz
RG           68K
SSB          0
WDW          EM
GB           0
MB           1.60
PC           1.40
  
```

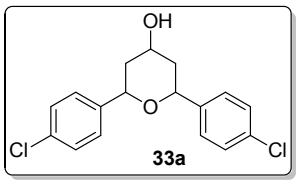
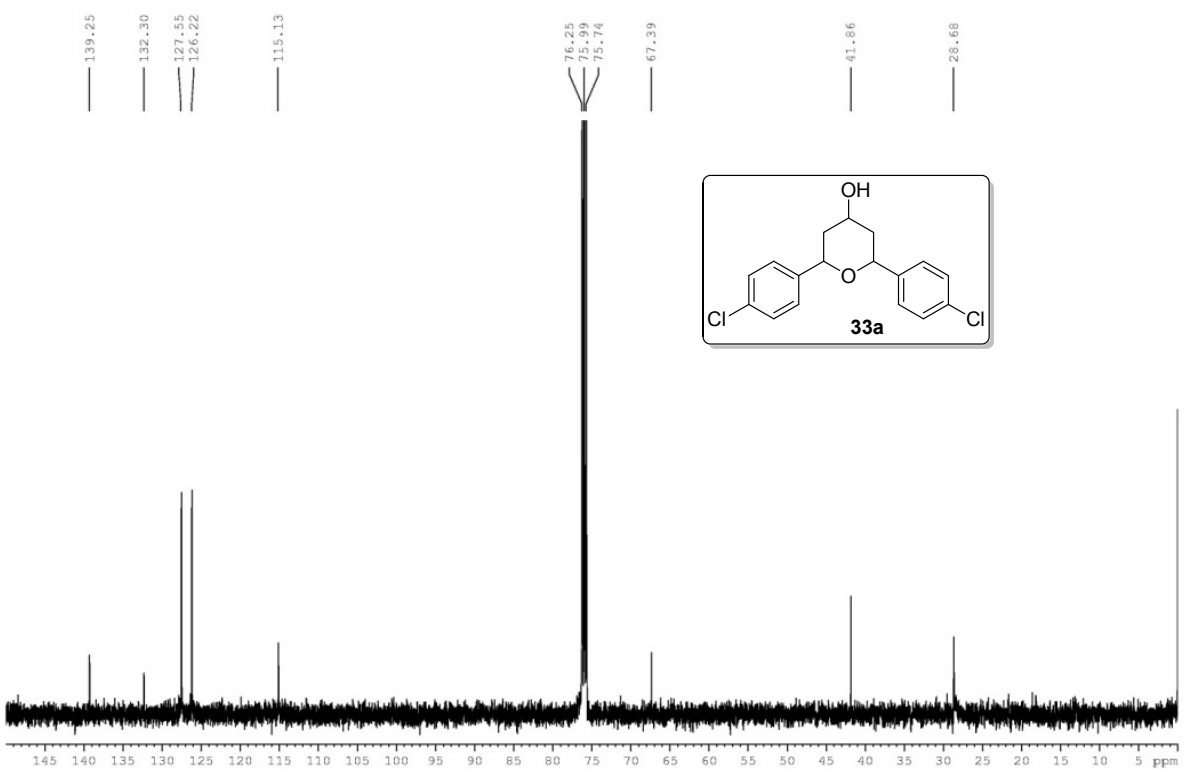
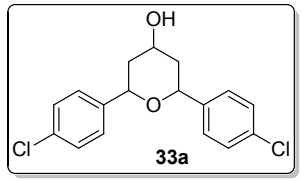
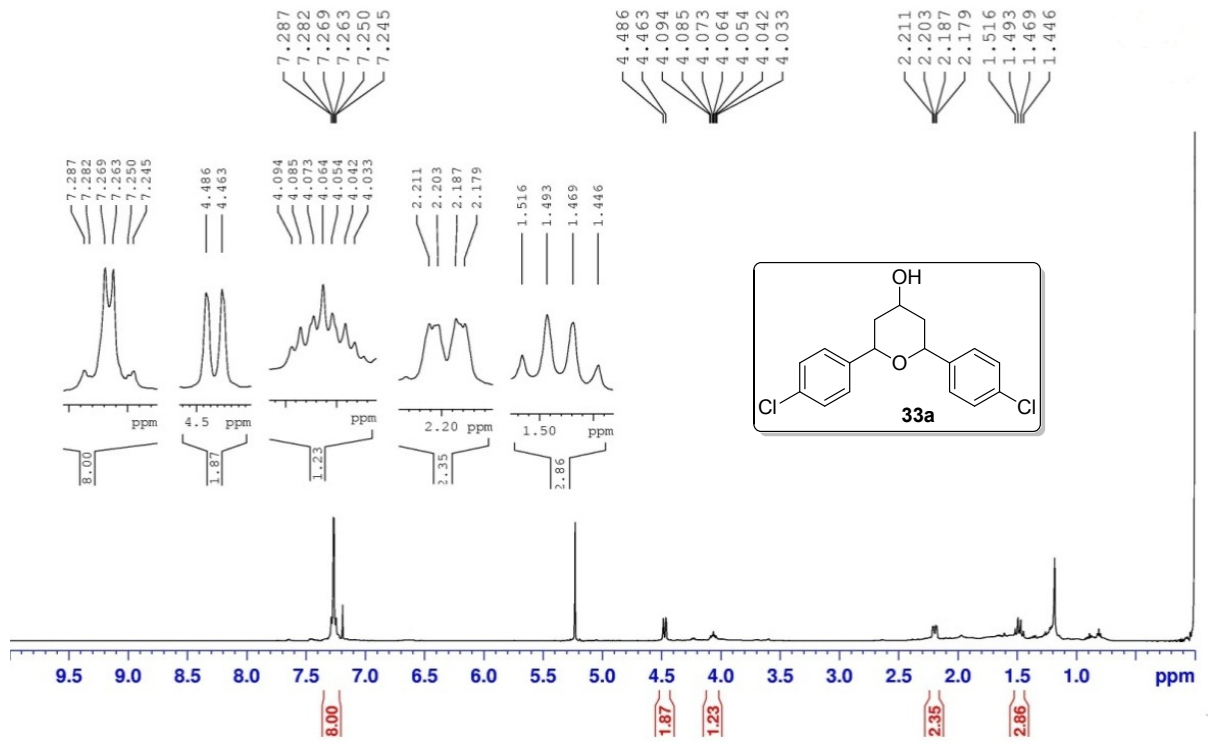


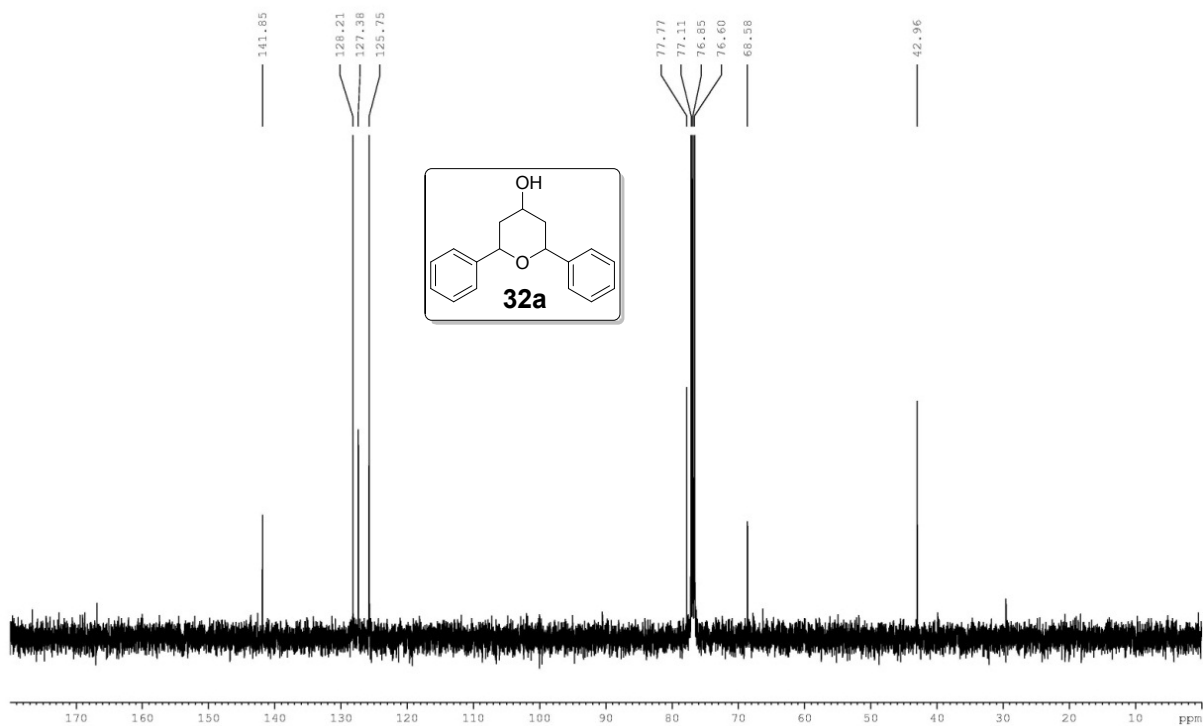
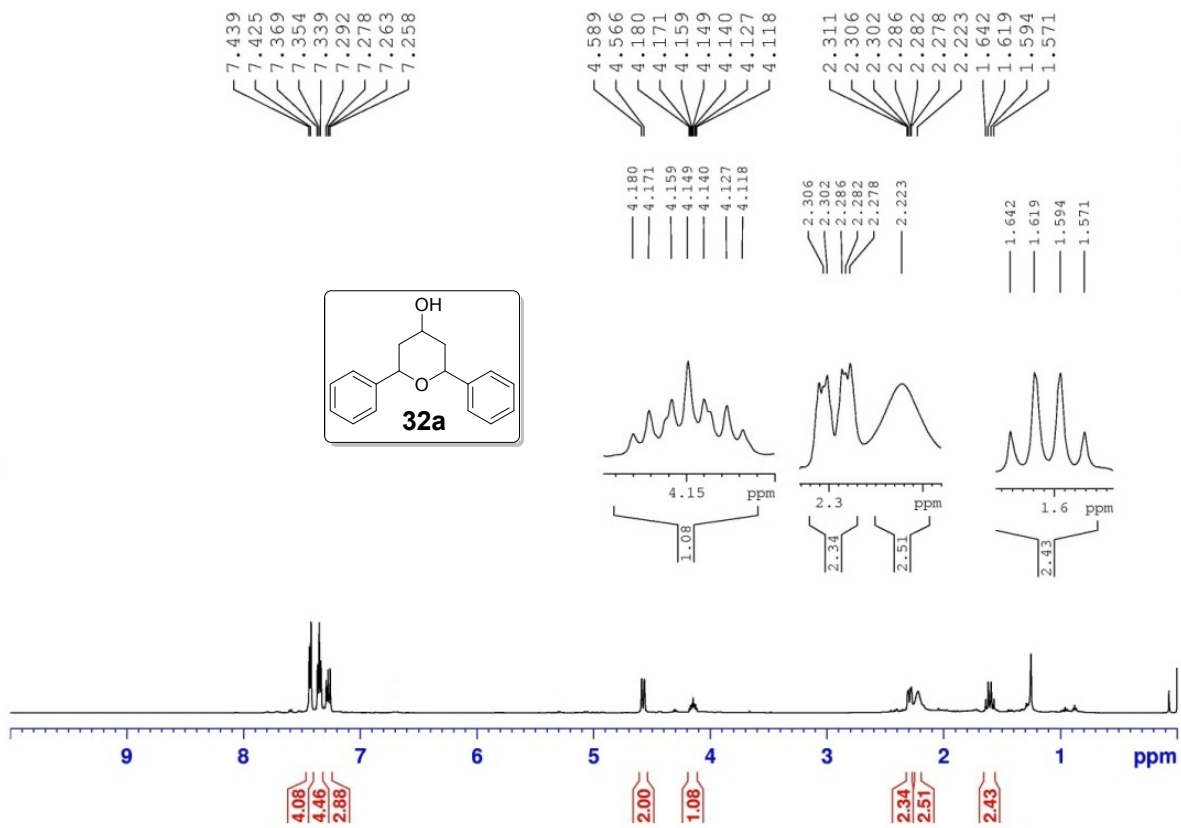












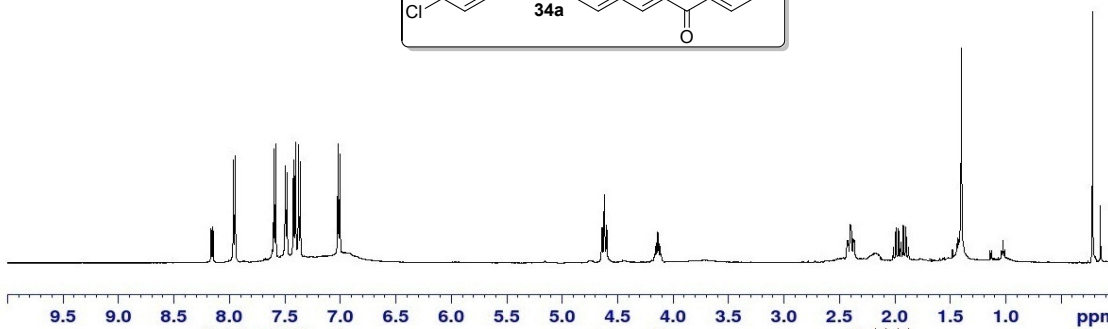
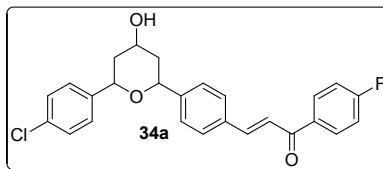
8
PROTON CDC13

8.185
8.179
7.962
7.946
7.599
7.582
7.495
7.479
7.421
7.404
7.401
7.376
7.359
7.021
7.003

4.173
4.164
4.151
4.173
4.164
4.151
4.141
4.132
4.119
4.110
2.285
2.281
2.276
2.255
2.250
2.229
2.224
2.220
2.046
1.869
1.845
1.821
1.801
1.798
1.778
1.754
1.731



```
Current Data Parameters
NAME      RMR-2-172
EXPNO    2
PROCNO   1
F2 - Acquisition Parameters
Date_    20120704
Time     15.24
INSTRUM  spect
PROBHD   5 mm PABBO BBO-
PULPROG  zgpg30
TD       65536
SOLVENT  CDCl3
NS       153
DS       2
SWH      30030.029 H
FIDRES   0.458222 H
AQ       1.0912244 s
RG       1620
DM       16.650 u
DE       6.00 u
TE       297.3 K
D1       2.0000000 s
d11      0.0300000 s
DELTA    1.8999999 s
TDO      1
```



1.00
2.00
3.02
2.17
2.32
2.06
2.15
2.00
1.00
2.15
1.00
2.32

188.84

166.72
164.69
144.64
143.77
140.89
134.59
134.39
131.25
130.06
128.72
127.71
126.55
125.97
121.68
115.94

78.05
77.70
77.46
77.20
76.95
69.46

40.05



```
Current Data Parameters
NAME      RMR-2-172
EXPNO    2
PROCNO   1
F2 - Acquisition Parameters
Date_    20120704
Time     15.24
INSTRUM  spect
PROBHD   5 mm PABBO BBO-
PULPROG  zgpg30
TD       65536
SOLVENT  CDCl3
NS       153
DS       2
SWH      30030.029 H
FIDRES   0.458222 H
AQ       1.0912244 s
RG       1620
DM       16.650 u
DE       6.00 u
TE       297.3 K
D1       2.0000000 s
d11      0.0300000 s
DELTA    1.8999999 s
TDO      1
===== CHANNEL f1 =====
NUC1     13C
P1       9.00 u
PL1     125.7703640 H
===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2     1H
PCPD2    80.00 u
PL2     18.40 H
PL12    20.00 G
PL13    20.00 G
SFO2    500.1320000 H
F2 - Processing parameter
SI       32768
SF       125.7573513 H
WDM     FM
SBB     0
LB      1.00 H
GB      0
PC      1.40
```

