Organocatalytic Asymmetric Domino *sulfa*-Michael-Aldol Reactions of 2-Mercaptobenzaldehyde with α,β-Unsaturated *N*-Acylpyrazoles for the Construction of Thiochromanes

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Supporting Information

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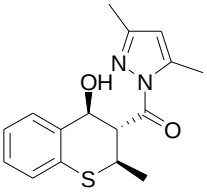
I. General Remarks

^{1}H NMR spectra were recorded on a VARIAN Mercury 300 MHz spectrometer in chloroform-d. Chemical shifts are reported in ppm with the internal TMS signal at 0.0 ppm as a standard. The data are reported as (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet or unresolved, brs = broad singlet, coupling constant(s) in Hz, integration). ^{13}C NMR spectra were recorded on a VARIAN Mercury 75 MHz spectrometer in chloroform-d. Chemical shifts are reported in ppm with the internal chloroform signal at 77.0 ppm as a standard. Commercially obtained reagents were used without further purification. All reactions were monitored by TLC with silica gel-coated plates. Enantiomeric ratios were determined by HPLC, using a chiralpak AS-H column, a chiralpak AD-H column, a chiralpak OD-H column, a chiralpak OJ-H column with hexane and *i*-PrOH as solvents. β-Aryl and β-alkyl α,β-unsaturated acids and *N*-acylpyrazoles were prepared according to the literatures.[1,2] The racemic adducts were obtained by using racemic catalyst **II** (0.1 eq.). The absolute (2*S*,3*R*,4*S*)-configuration of **3r** was determined by X-ray crystallographic analysis of a single crystal, the other adducts were deduced on the basis of these results.

II. General procedure for asymmetric domino *sulfa*-Michael-Aldol reactions of 2-mercaptobenzaldehyde with α,β-unsaturated *N*-acylpyrazoles catalyzed by organocatalysts (III)

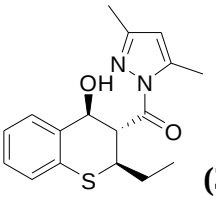
To a vial containing 2-mercaptobenzaldehyde (0.3 mmol) and catalyst **III** (1.7 mg, 0.003 mmol) in ether (1.0 mL) was added α,β-unsaturated *N*-acylpyrazoles (0.33 mmol) at 0^{o}C. TLC analysis indicated completion of the reactions after about 4h. Then the reaction mixtures were concentrated *in vacuo* to obtain the crude products. The crude products were purified by flash silica gel chromatography to afford the products.



(3a)

(2*S*,3*R*,4*S*)-3,5-Dimethyl-pyrazol-1-yl)-(4-hydroxy-2-methyl-thiochroman-3-yl)-methanone (Table 2, entry 1):

The title compound was prepared according to the general procedure as described above in 90% yield. $[\alpha]^{25}_{D}$ +135.8 (*c* 1.24, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 1.29-1.31 (d, *J* = 6.9 Hz, 3H), 2.21 (s, 3H), 2.61 (s, 3H), 3.80-3.88 (m, 1H), 4.22-4.35 (m, 1H), 4.96-5.00 (m, 1H), 6.03 (s, 1H), 7.02-7.18 (m, 3H), 7.65-7.67 (m, 1H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 13.7, 14.5, 19.8, 37.2, 53.4, 71.9, 111.9, 124.5, 125.5, 126.2, 127.6, 132.6, 136.8, 144.5, 152.9, 173.7; IR (KBr) ν: 3476, 2959, 2925, 2169, 1699, 1394, 1364, 1331, 1296, 1220, 1125, 1057, 765 cm^{-1}. The product was analyzed to determine the diastereoselectivity and enantioselectivity of the reaction: dr = 98:2, 98% ee (major diastereomer) determined by HPLC (Chiralcel AS-H, i-propanol/hexane = 10/90, flow rate 0.5 mL/min, λ = 254 nm); major diastereomer: t_{minor} = 22.1 min, t_{major} = 15.1 min; minor diastereomer: 14.5 min, 23.5 min.

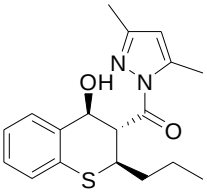


(3b)

(2*S*,3*R*,4*S*)-3,5-Dimethyl-pyrazol-1-yl)-(2-ethyl-4-hydroxy-thiochroman-3-yl)-methanone (Table 2, entry 2):

The title compound was prepared according to the general procedure as described above in 88% yield. $[\alpha]^{25}_{D}$ +128.7 (*c* 1.26 $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 0.94-0.99 (t, *J* = 7.2 Hz, 3H), 1.51-1.77 (m, 2H), 2.21 (s, 3H), 2.60 (s, 3H), 3.05 (brs, 1H), 3.76-3.84 (m, 1H), 4.25-4.31 (t, *J* = 10.2 Hz, 1H), 4.93-4.96 (d, *J* = 9.6 Hz, 1H), 6.02 (s, 1H), 7.16-7.19 (m, 3H), 7.63-7.66 (m, 1H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 10.8, 13.7, 14.5, 27.6, 44.6, 52.2, 72.0, 111.8, 124.6, 125.5, 126.2, 127.5,

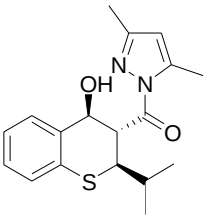
132.2, 137.7, 144.5, 152.9, 173.8; IR (KBr) ν: 3464, 2965, 2927, 2166, 1700, 1588, 1437, 1396, 1361, 1318, 1261, 1219, 1200, 1125, 1037, 1019, 961, 768, 747 cm^{-1}. The product was analyzed to determine the diastereoselectivity and enantioselectivity of the reaction: dr = 97:3, 96% ee (major diastereomer) determined by HPLC (Chiralcel AD-H, i-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 254 nm); major diastereomer: t_{minor} = 8.1 min, t_{major} = 14.2 min; minor diastereomer: 11.8 min, 12.6 min.



(3c)

(2*S*,3*R*,4*S*)-(3,5-Dimethyl-pyrazol-1-yl)-(4-hydroxy-2-propyl-thiochroman-3-yl)-methanone (Table 2, entry 3):

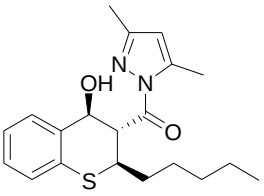
The title compound was prepared according to the general procedure as described above in 91% yield. $[\alpha]^{25}_{D}$ +133.8 (*c* 1.18 $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 0.84-0.89 (t, *J* = 7.2 Hz, 3H), 1.25-1.67 (m, 4H), 2.21 (s, 3H), 2.61 (s, 3H), 3.81-3.89 (m, 1H), 4.24-4.31 (t, *J* = 10.2 Hz, 1H), 4.93-4.96 (d, *J* = 9.6 Hz, 1H), 6.03 (s, 1H), 7.16-7.19 (m, 3H), 7.64-7.66 (m, 1H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 13.6, 13.7, 14.5, 19.5, 36.7, 42.8, 52.6, 72.0, 111.8, 124.6, 125.5, 126.2, 127.5, 132.2, 137.7, 144.5, 152.9, 173.8; IR (KBr) ν: 3471, 2954, 2925, 2176, 1701, 1588, 1474, 1437, 1382, 1359, 1333, 1299, 1219, 1200, 1034, 961, 768, 750 cm^{-1}. The product was analyzed to determine the diastereoselectivity and enantioselectivity of the reaction: dr = 97:3, 97% ee (major diastereomer) determined by HPLC (Chiralcel OJ-H, i-propanol/hexane = 10/90, flow rate 0.5 mL/min, λ = 254 nm); major diastereomer: t_{minor} = 13.2 min, t_{major} = 12.0 min; minor diastereomer: 20.3 min, 26.8 min.



(3d)

(2*S*,3*R*,4*S*)-(3,5-Dimethyl-pyrazol-1-yl)-(4-hydroxy-2-isopropyl-thiochroman-3-yl)-methanone (Table 2, entry 4):

The title compound was prepared according to the general procedure as described above in 91% yield. $[\alpha]^{25}_{D}$ +106.1 (*c* 1.30 $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 0.87-0.89 (d, *J* = 6.6 Hz, 3H), 0.96-0.98 (d, *J* = 6.6 Hz, 3H), 1.90-1.98 (m, 1H), 2.21 (s, 3H), 2.60 (s, 3H), 3.06 (brs, 1H), 3.96-3.99 (m, 1H), 4.25-4.32 (t, *J* = 10.2 Hz, 1H), 4.80-4.95 (m, 1H), 6.02 (s, 1H), 7.20-7.21 (m, 3H), 7.60-7.70 (m, 1H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 13.8, 14.6, 16.7, 20.6, 31.5, 50.0, 50.6, 72.6, 111.8, 124.7, 124.8, 126.8, 127.6, 132.7, 139.0, 144.6, 153.0, 173.9; IR (KBr) ν: 3444, 2961, 2930, 2170, 1718, 1586, 1467, 1437, 1408, 1378, 1359, 1339, 1306, 1219, 1131, 1084, 1036, 965, 765 cm^{-1}. The product was analyzed to determine the diastereoselectivity and enantioselectivity of the reaction: dr = 93:7, 91% ee (major diastereomer) determined by HPLC (Chiralcel AD-H, i-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 254 nm); major diastereomer: t_{minor} = 8.2 min, t_{major} = 13.1 min; minor diastereomer: 10.6 min, 11.5 min.

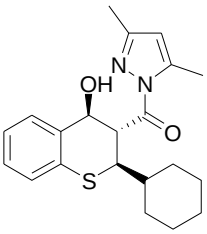


(3e)

(2*S*,3*R*,4*S*)-(3,5-Dimethyl-pyrazol-1-yl)-(4-hydroxy-2-pentyl-thiochroman-3-yl)-methanone (Table 2, entry 5):

The title compound was prepared according to the general procedure as described above in 85% yield. $[\alpha]^{25}_{D}$ +126.8 (*c* 1.47 $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 0.82-0.86 (t, *J* = 6.6 Hz, 3H), 1.22-1.70 (m, 8H), 2.21 (s, 3H), 2.61 (s, 3H), 3.06 (brs, 1H), 3.80-3.85 (m, 1H), 4.25-4.30 (t, *J* = 9.6 Hz, 1H), 4.93-4.97 (d, *J* = 10.5 Hz,

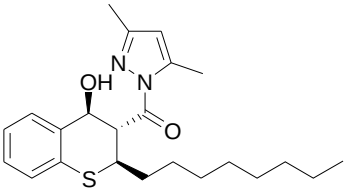
1H), 6.03 (s, 1H), 7.16-7.19 (m, 3H), 7.64-7.67 (m, 1H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 13.7, 13.9, 14.5, 22.3, 25.9, 31.2, 34.5, 43.0, 52.6, 72.0, 111.8, 124.7, 125.6, 126.2, 127.5, 132.3, 137.6, 144.5, 152.9, 173.9; IR (KBr) ν: 3474, 2955, 2929, 2857, 1708, 1587, 1566, 1469, 1436, 1383, 1361, 1339, 1302, 1261, 1224, 1201, 1130, 1087, 1057, 1037, 961, 901, 746 cm^{-1}. The product was analyzed to determine the diastereoselectivity and enantioselectivity of the reaction: dr = 96:4, 97% ee (major diastereomer) determined by HPLC (Chiralcel OJ-H, i-propanol/hexane = 10/90, flow rate 0.5 mL/min, λ = 254 nm); major diastereomer: t_{minor} = 10.3 min, t_{major} = 9.5 min; minor diastereomer: 11.8 min, 14.8 min.



(3f)

(2*S*,3*R*,4*S*)-(2-Cyclohexyl-4-hydroxy-thiochroman-3-yl)-(3,5-dimethyl-pyrazol-1-yl)-methanone (Table 2, entry 6):

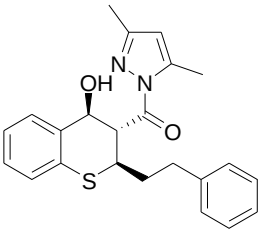
The title compound was prepared according to the general procedure as described above in 91% yield. $[\alpha]^{25}_{D}$ +72.5 (*c* 0.6 $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 0.82-1.70 (m, 11H), 2.21 (s, 3H), 2.60 (s, 3H), 3.20 (brs, 1H), 3.88-3.93 (m, 1H), 4.25-4.32 (t, *J* = 10.2 Hz, 1H), 4.86-4.90 (d, *J* = 9.6 Hz, 1H), 6.02 (s, 1H), 7.18-7.28 (m, 3H), 7.61-7.65 (m, 1H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 13.8, 14.5, 26.0, 26.1, 26.4, 27.5, 30.8, 41.9, 49.4, 50.3, 72.7, 111.7, 124.6, 125.0, 126.9, 127.6, 132.8, 139.3, 144.7, 153.0, 173.8; IR (KBr) ν: 3421, 2925, 2852, 2166, 1718, 1586, 1558, 1435, 1408, 1379, 1362, 1324, 1306, 1220, 1128, 1087, 1057, 1037, 963, 901, 772, 750 cm^{-1}. The product was analyzed to determine the diastereoselectivity and enantioselectivity of the reaction: dr = 96:4, 93% ee (major diastereomer) determined by HPLC (Chiralcel AD-H, i-propanol/hexane = 5/95, flow rate 0.25 mL/min, λ = 235 nm); major diastereomer: t_{minor} = 70.9 min, t_{major} = 75.7 min; minor diastereomer: 87.1 min.



(3g)

(2*S*,3*R*,4*S*)-(3,5-Dimethyl-pyrazol-1-yl)-(4-hydroxy-2-octyl-thiochroman-3-yl)-methanone (Table 2, entry 7):

The title compound was prepared according to the general procedure as described above in 89% yield. $[\alpha]^{25}_{D}$ +105.2 (*c* 0.76 $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 0.84-0.88 (t, *J* = 6.9 Hz, 3H), 1.21-1.66 (m, 14H), 2.20 (s, 3H), 2.60 (s, 3H), 3.79-3.87 (m, 1H), 4.24-4.31 (t, *J* = 10.2 Hz, 1H), 4.93-4.96 (d, *J* = 10.2 Hz, 1H), 6.02 (s, 1H), 7.16-7.18 (m, 3H), 7.64-7.66 (m, 1H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 13.9, 14.2, 14.6, 22.7, 26.3, 29.2, 29.4, 31.9, 34.7, 43.1, 52.8, 72.2, 111.9, 124.8, 125.7, 126.4, 127.7, 132.4, 137.8, 144.6, 153.0, 174.0; IR (KBr) ν: 3493, 2952, 2921, 2853, 1712, 1586, 1563, 1472, 1383, 1356, 1262, 1037, 961, 757 cm^{-1}. The product was analyzed to determine the diastereoselectivity and enantioselectivity of the reaction: dr = 98:2, 98% ee (major diastereomer) determined by HPLC (Chiralcel AD-H, i-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 254 nm); major diastereomer: t_{minor} = 5.8 min, t_{major} = 7.7 min; minor diastereomer: 7.0 min, 9.3 min.

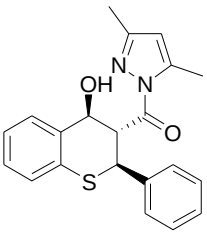


(3h)

(2*S*,3*R*,4*S*)-(3,5-Dimethyl-pyrazol-1-yl)-(4-hydroxy-2-phenethyl-thiochroman-3-yl)-methanone (Table 2, entry 8):

The title compound was prepared according to the general procedure as described above in 93% yield. $[\alpha]^{25}_{D}$ +97.5 (*c* 1.59 $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 1.80-1.85 (m, 1H), 1.97-2.02 (m, 1H), 2.19 (s, 3H), 2.58 (s, 3H), 2.64-2.68 (m, 1H), 2.77-2.82 (m, 1H), 3.10 (brs, 1H), 3.82-3.86 (m, 1H), 4.26-4.33 (t, *J* = 9.9 Hz, 1H), 4.94-4.98 (d, *J* = 9.9 Hz, 1H), 6.01 (s, 1H), 7.08-7.24 (m, 8H), 7.63-7.66 (m, 1H); ^{13}C

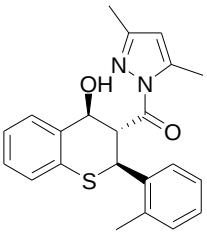
NMR ($CDCl_3$, TMS, 75 MHz) δ 13.7, 14.5, 32.4, 36.5, 42.7, 52.7, 71.8, 111.9, 125.0, 125.5, 125.9, 126.5, 127.6, 128.3, 131.9, 137.9, 141.0, 144.5, 152.9, 173.6; IR (KBr) ν: 3493, 2926, 2856, 1706, 1588, 1566, 1471, 1434, 1384, 1362, 1344, 1201, 1180, 1131, 1080, 1048, 996, 962, 896, 812, 751 cm^{-1}. The product was analyzed to determine the diastereoselectivity and enantioselectivity of the reaction: dr = 94:6, 98% ee (major diastereomer) determined by HPLC (Chiralcel AS-H, i-propanol/hexane = 10/90, flow rate 0.5 mL/min, λ = 254 nm); major diastereomer: t_{minor} = 22.9 min, t_{major} = 16.0 min; minor diastereomer: 18.1 min, 20.0 min.



(3i)

(2*S*,3*R*,4*S*)-(3,5-Dimethyl-pyrazol-1-yl)-(4-hydroxy-2-phenyl-thiochroman-3-yl)-methanone (Table 3, entry 1):

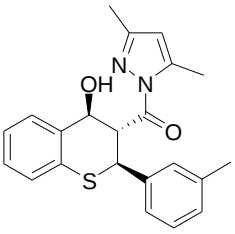
The title compound was prepared according to the general procedure as described above in 87% yield. $[\alpha]^{25}_{D}$ +105.2 (*c* 2.0 $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 2.18 (s, 3H), 2.28 (s, 3H), 4.90-4.98 (m, 2H), 5.12-5.15 (d, *J* = 9.3 Hz, 1H), 5.86 (s, 1H), 7.13-7.26 (m, 6H), 7.34-7.36 (m, 2H), 7.72-7.75 (m, 1H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 13.6, 14.0, 46.8, 52.3, 72.2, 111.3, 124.6, 125.0, 126.5, 127.7, 128.0, 128.2, 128.4, 133.0, 136.7, 137.7, 144.2, 152.6, 172.8; IR (KBr) ν: 3493, 2926, 1706, 1588, 1471, 1434, 1384, 1362, 1344, 1201, 1180, 1131, 1048, 996, 962, 896, 812, 751 cm^{-1}. The product was analyzed to determine the diastereoselectivity and enantioselectivity of the reaction: dr = 99:1, 99% ee (major diastereomer) determined by HPLC (Chiralcel OJ-H, i-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 254 nm); major diastereomer: t_{minor} = 13.2 min, t_{major} = 16.9 min; minor diastereomer: 25.8 min, 30.2 min.



(3j)

(2*S*,3*R*,4*S*)-(3,5-Dimethyl-pyrazol-1-yl)-(4-hydroxy-2-*o*-tolyl-thiochroman-3-yl)-methanone (Table 3, entry 2):

The title compound was prepared according to the general procedure as described above in 85% yield. $[\alpha]^{25}_{D}$ +120.8 (*c* 1.28 $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 2.21 (s, 3H), 2.26 (s, 3H), 2.49 (s, 3H), 3.30 (brs, 1H), 5.04-5.08 (m, 1H), 5.15-5.22 (m, 2H), 5.89 (s, 1H), 7.01-7.32 (m, 7H), 7.73-7.80 (m, 1H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 13.7, 13.9, 19.4, 42.2, 51.7, 72.6, 111.3, 124.6, 125.1, 126.2, 126.9, 127.7, 127.8, 130.5, 133.2, 135.8, 136.6, 144.2, 152.7, 173.1; IR (KBr) ν: 3421, 3061, 2926, 2360, 2342, 1718, 1586, 1489, 1465, 1437, 1384, 1361, 1338, 1301, 1262, 1218, 1161, 1130, 1037, 992, 901, 772, 747 cm^{-1}. The product was analyzed to determine the diastereoselectivity and enantioselectivity of the reaction: dr = 98:2, 99% ee (major diastereomer) determined by HPLC (Chiralcel AD-H, i-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 254 nm); major diastereomer: t_{minor} = 9.1 min, t_{major} = 22.1 min; minor diastereomer: 17.5 min, 18.2 min.

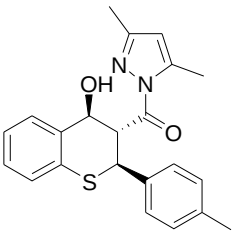


(3k)

(2*S*,3*R*,4*S*)-(3,5-Dimethyl-pyrazol-1-yl)-(4-hydroxy-2-*m*-tolyl-thiochroman-3-yl)-methanone (Table 3, entry 3):

The title compound was prepared according to the general procedure as described above in 88% yield. $[\alpha]^{25}_{D}$ +92.0 (*c* 1.39 $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 2.19 (s, 3H), 2.24 (s, 3H), 2.29 (s, 3H), 3.20 (brs, 1H), 4.90-4.93 (m, 2H), 5.10-5.20 (m, 1H), 5.87 (s, 1H), 6.98-7.00 (m, 1H), 7.06-7.23 (m, 6H), 7.72-7.75 (m, 1H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 13.7, 14.0, 21.2, 46.8, 52.2, 72.2, 111.2, 124.6,

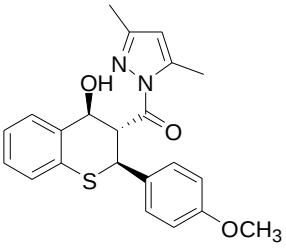
125.0, 125.1, 126.5, 127.7, 128.2, 128.8, 129.0, 133.1, 136.7, 137.5, 138.0, 144.2, 152.5, 172.9; IR (KBr) ν: 3421, 3058, 2925, 2360, 2337, 1719, 1606, 1588, 1566, 1472, 1436, 1384, 1361, 1336, 1302, 1262, 1228, 1170, 1130, 1078, 1037, 962, 898, 787, 749 cm^{-1}. The product was analyzed to determine the diastereoselectivity and enantioselectivity of the reaction: dr = 97:3, 99% ee (major diastereomer) determined by HPLC (Chiralcel AD-H, i-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 254 nm); major diastereomer: t_{minor} = 10.7 min, t_{major} = 26.8 min; minor diastereomer: 21.0 min, 22.1 min.



(3l)

(2*S*,3*R*,4*S*)-(3,5-Dimethyl-pyrazol-1-yl)-(4-hydroxy-2-*p*-tolyl-thiochroman-3-yl)-methanone (Table 3, entry 4):

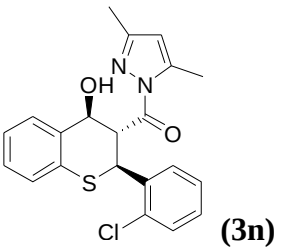
The title compound was prepared according to the general procedure as described above in 89% yield. $[\alpha]^{25}_{D}$ +115.5 (*c* 1.42 $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 2.19 (s, 3H), 2.26 (s, 3H), 2.31 (s, 3H), 3.20 (brs, 1H), 4.89-4.97 (m, 2H), 5.08-5.18 (m, 1H), 5.88 (s, 1H), 7.01-7.04 (m, 2H), 7.15-7.26 (m, 5H), 7.72-7.74 (m, 1H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 13.8, 14.1, 21.1, 46.5, 52.4, 72.4, 111.4, 124.6, 125.1, 126.6, 127.8, 128.1, 129.2, 133.2, 134.9, 136.9, 137.8, 144.3, 152.7, 172.9; IR (KBr) ν: 3426, 3050, 2924, 2852, 2164, 1719, 1588, 1512, 1437, 1411, 1379, 1361, 1309, 1261, 1037, 962, 898, 805, 749 cm^{-1}. The product was analyzed to determine the diastereoselectivity and enantioselectivity of the reaction: dr = 98:2, 98% ee (major diastereomer) determined by HPLC (Chiralcel AD-H, i-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 254 nm); major diastereomer: t_{minor} = 10.9 min, t_{major} = 26.0 min; minor diastereomer: 22.8 min.



(3m)

(2*S*,3*R*,4*S*)-(3,5-Dimethyl-pyrazol-1-yl)-[4-hydroxy-2-(4-methoxy-phenyl)-thio-chroman-3-yl]-methanone (Table 3, entry 5):

The title compound was prepared according to the general procedure as described above in 86% yield. $[\alpha]^{25}_{D}$ +106.1 (*c* 1.66 $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 2.19 (s, 3H), 2.31 (s, 3H), 3.75 (s, 3H), 4.85-4.97 (m, 2H), 5.10-5.14 (m, 1H), 5.88 (s, 1H), 6.73-6.76 (d, *J* = 8.7 Hz, 2H), 7.14-7.19 (m, 3H), 7.26-7.30 (m, 2H), 7.72-7.74 (m, 1H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 13.6, 14.0, 46.2, 52.3, 55.0, 72.3, 111.3, 113.7, 124.5, 124.9, 126.5, 127.7, 129.3, 129.6, 133.1, 136.6, 144.1, 152.5, 159.1, 172.9; IR (KBr) ν: 3445, 2960, 2928, 2836, 1716, 1610, 1585, 1512, 1464, 1438, 1379, 1360, 1305, 1252, 1176, 1130, 1035, 962, 990, 817, 749 cm^{-1}. The product was analyzed to determine the diastereoselectivity and enantioselectivity of the reaction: dr = 97:3, 97% ee (major diastereomer) determined by HPLC (Chiralcel AD-H, i-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 254 nm); major diastereomer: t_{minor} = 17.1 min, t_{major} = 39.5 min; minor diastereomer: 30.0 min, 33.7 min.

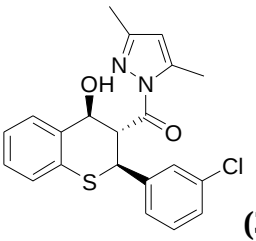


(3n)

(2*S*,3*R*,4*S*)-[2-(2-Chloro-phenyl)-4-hydroxy-thiochroman-3-yl]-(3,5-dimethyl-pyrazol-1-yl)-methanone (Table 3, entry 6):

The title compound was prepared according to the general procedure as described above in 90% yield. $[\alpha]^{25}_{D}$ +57.4 (*c* 1.51 $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 2.18 (s, 3H), 2.34 (s, 3H), 3.33-3.36 (brs, 1H), 4.93-5.00 (t, *J* = 10.2 Hz, 1H), 5.10-5.20 (m, 1H), 5.51-5.54 (d, *J* = 10.8 Hz, 1H), 5.91 (s, 1H), 7.09-7.26 (m, 5H),

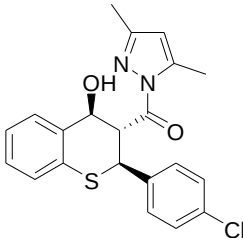
7.31-7.37 (m, 2H), 7.74-7.75 (m, 1H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 13.7, 14.2, 42.5, 51.8, 72.3, 111.5, 125.1, 125.6, 126.5, 127.1, 127.8, 128.7, 128.9, 129.7, 132.6, 134.0, 136.1, 137.2, 144.5, 152.9, 172.6; IR (KBr) ν: 3509, 2960, 2928, 2836, 1701, 1590, 1383, 1362, 1335, 1022, 961, 749 cm^{-1}. The product was analyzed to determine the diastereoselectivity and enantioselectivity of the reaction: dr = 97:3, 97% ee (major diastereomer) determined by HPLC (Chiralcel AD-H, i-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 254 nm); major diastereomer: t_{minor} = 13.9 min, t_{major} = 38.3 min; minor diastereomer: 25.1 min, 33.8 min.



(3o)

(2*S*,3*R*,4*S*)-[2-(3-Chloro-phenyl)-4-hydroxy-thiochroman-3-yl]-(3,5-dimethyl-pyrazol-1-yl)-methanone (Table 3, entry 7):

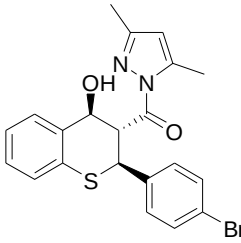
The title compound was prepared according to the general procedure as described above in 92% yield. $[\alpha]^{25}_{D}$ +119.1 (*c* 1.03 $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 2.18 (s, 3H), 2.32 (s, 3H), 3.22-3.28 (brs, 1H), 4.80-4.88 (m, 2H), 5.11-5.16 (m, 1H), 5.87 (s, 1H), 7.12-7.26 (m, 6H), 7.40 (s, 1H), 7.70-7.73 (m, 1H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 13.4, 13.8, 46.2, 52.1, 71.8, 111.3, 124.7, 125.0, 126.2, 127.7, 128.0, 128.3, 129.3, 132.2, 133.9, 136.5, 139.8, 144.1, 152.7, 172.3; IR (KBr) ν: 3509, 2926, 2850, 2362, 1704, 1590, 1473, 1383, 1362, 1335, 1294, 1265, 1214, 1127, 1078, 1022, 961, 901, 818, 789, 748 cm^{-1}. The product was analyzed to determine the diastereoselectivity and enantioselectivity of the reaction: dr = 95:5, 98% ee (major diastereomer) determined by HPLC (Chiralcel AD-H, i-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 254 nm); major diastereomer: t_{minor} = 13.3 min, t_{major} = 43.3 min; minor diastereomer: 26.0 min, 30.0 min.



(3p)

(2*S*,3*R*,4*S*)-[2-(4-Chloro-phenyl)-4-hydroxy-thiochroman-3-yl]-(3,5-dimethyl-pyrazol-1-yl)-methanone (Table 3, entry 8):

The title compound was prepared according to the general procedure as described above in 90% yield. $[\alpha]^{25}_{D}$ +111.9 (*c* 1.45 $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 2.18 (s, 3H), 2.34 (s, 3H), 4.84-4.96 (m, 2H), 5.09-5.12 (d, *J* = 9.6 Hz, 1H), 5.90 (s, 1H), 7.16-7.21 (m, 5H), 7.29-7.32 (m, 2H), 7.72-7.75 (m, 1H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 13.7, 14.2, 46.1, 52.4, 72.2, 111.6, 124.9, 125.2, 126.5, 127.9, 128.7, 129.7, 132.7, 133.8, 136.9, 144.4, 153.0, 172.5; IR (KBr) ν: 3494, 2925, 2853, 1702, 1586, 1491, 1473, 1435, 1383, 1361, 1336, 1294, 1218, 1130, 1087, 1025, 961, 771, 747 cm^{-1}. The product was analyzed to determine the diastereoselectivity and enantioselectivity of the reaction: dr = 98:2, 99% ee (major diastereomer) determined by HPLC (Chiralcel AD-H, i-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 254 nm); major diastereomer: t_{minor} = 11.4 min, t_{major} = 26.0 min; minor diastereomer: 20.8 min.

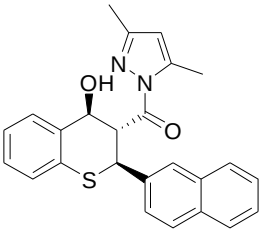


(3q)

(2*S*,3*R*,4*S*)-[2-(4-Bromo-phenyl)-4-hydroxy-thiochroman-3-yl]-(3,5-dimethyl-pyrazol-1-yl)-methanone (Table 3, entry 9):

The title compound was prepared according to the general procedure as described above in 94% yield. $[\alpha]^{25}_{D}$ +108.4 (*c* 1.79 $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 2.18 (s, 3H), 2.34 (s, 3H), 3.18 (brs, 1H), 4.84-4.91 (m, 2H), 5.06-5.12 (m, 1H), 5.91 (s, 1H), 7.15-7.36 (m, 7H), 7.70-7.73 (m, 1H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 13.8, 14.2, 46.2, 52.5, 72.3, 111.7, 125.0, 125.2, 126.4, 128.0, 130.0, 131.7, 137.4,

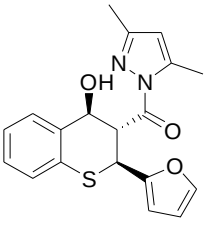
144.5, 150.3, 152.5, 172.5; IR (KBr) ν: 3490, 2924, 2898, 1701, 1586, 1488, 1472, 1434, 1385, 1361, 1336, 1290, 1218, 1130, 1073, 1025, 1010, 962, 809, 747 cm^{-1}. The product was analyzed to determine the diastereoselectivity and enantioselectivity of the reaction: dr = 98:2, 97% ee (major diastereomer) determined by HPLC (Chiralcel AD-H, i-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 254 nm); major diastereomer: t_{minor} = 11.9 min, t_{major} = 26.0 min; minor diastereomer: 22.5 min.



(3r)

(2*S*,3*R*,4*S*)-(3,5-Dimethyl-pyrazol-1-yl)-(4-hydroxy-2-naphthalen-2-yl-thio-chroman-3-yl)-methanone (Table 3, entry 10):

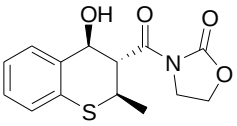
The title compound was prepared according to the general procedure as described above in 87% yield. $[\alpha]^{25}_{D}$ +89.6 (*c* 1.43 $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 2.16 (s, 3H), 2.22 (s, 3H), 3.01 (brs, 1H), 5.00-5.04 (m, 1H), 5.13-5.18 (m, 2H), 5.78 (s, 1H), 7.18-7.24 (m, 3H), 7.42-7.50 (m, 3H), 7.68-7.74 (m, 4H), 7.80-7.83 (m, 1H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 13.7, 14.1, 47.1, 52.4, 72.3, 111.4, 124.8, 125.2, 125.6, 126.1, 126.4, 127.5, 127.7, 127.9, 128.3, 133.0, 135.5, 137.0, 144.3, 152.7, 172.8; IR (KBr) ν: 3421, 3058, 2964, 2924, 2848, 2360, 2341, 1717, 1585, 1436, 1379, 1359, 1219, 1128, 1037, 962, 772 cm^{-1}. The product was analyzed to determine the diastereoselectivity and enantioselectivity of the reaction: dr = 97:3, 99% ee (major diastereomer) determined by HPLC (Chiralcel AD-H, i-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 254 nm); major diastereomer: t_{minor} = 19.4 min, t_{major} = 38.7 min; minor diastereomer: 32.3 min.



(3s)

(2*S*,3*R*,4*S*)-(3,5-Dimethyl-pyrazol-1-yl)-(2-furan-2-yl-4-hydroxy-thiochroman-3-yl)-methanone (Table 3, entry 11):

The title compound was prepared according to the general procedure as described above in 91% yield. $[\alpha]^{25}_{D}$ +169.6 (*c* 0.6 $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 2.21 (s, 3H), 2.48 (s, 3H), 4.70-4.78 (t, *J* = 10.2 Hz, 1H), 4.99-5.03 (m, 1H), 5.16-5.19 (m, 1H), 5.98 (s, 1H), 6.14-6.20 (m, 2H), 7.20-7.26 (m, 4H), 7.70-7.72 (m, 1H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 13.5, 14.0, 39.4, 50.8, 71.6, 107.1, 110.1, 111.3, 125.0, 125.4, 125.9, 127.5, 131.6, 137.6, 142.1, 144.5, 151.6, 152.9, 172.1; IR (KBr) ν: 3480, 2959, 2925, 2853, 2360, 2341, 1706, 1587, 1473, 1438, 1383, 1362, 1261, 1222, 1201, 1165, 1131, 1037, 962, 749 cm^{-1}. The product was analyzed to determine the diastereoselectivity and enantioselectivity of the reaction: dr = 95:5, 95% ee (major diastereomer) determined by HPLC (Chiralcel AD-H, i-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 254 nm); major diastereomer: t_{minor} = 13.8 min, t_{major} = 23.2 min; minor diastereomer: 9.2 min, 9.7 min.

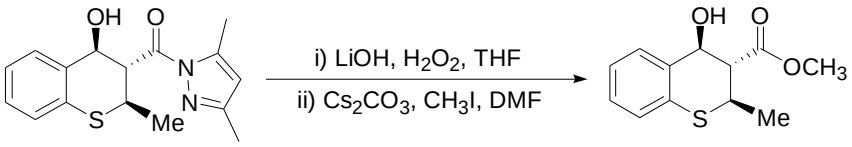


(3t)

(2*S*,3*R*,4*S*)- 3-(4-Hydroxy-2-methyl-thiochroman-3-carbonyl)-oxazolidin-2-one:

The title compound was prepared according to the general procedure as described above in 92% yield. ^{1}H NMR (d_6-DMSO, TMS, 300 MHz) δ 1.38-1.40 (d, *J* = 6.6 Hz, 3H), 4.15-4.24 (m, 3H), 4.41-4.48 (m, 1H), 4.50-5.58 (m, 2H), 4.90-4.94 (m, 1H), 6.30-6.32 (m, 1H), 7.26-7.32 (m, 2H), 7.68-7.71 (m, 1H); ^{13}C NMR (d_6-DMSO, TMS, 75 MHz) δ 20.0, 37.9, 43.5, 51.3, 62.8, 70.9, 125.0, 125.7, 127.3, 128.1, 132.5, 138.4, 154.3, 174.5. The product was analyzed to determine the diastereoselectivity and enantioselectivity of the reaction: dr = 91:9, 62% ee (major diastereomer) determined by HPLC (Chiralcel OD-H, i-propanol/hexane = 30/70, flow rate 1.0 mL/min, λ = 220 nm); major diastereomer: t_{minor} = 13.2 min, t_{major} = 10.7 min; minor diastereomer: 15.3 min, 22.1 min.

III. Synthetic transformation of the *sulfa*-Michael-Aldol adduct **3a**[3,4]



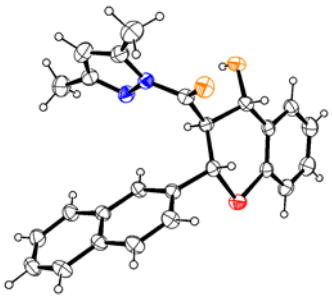
3a
98:2 dr, 98% ee

4a
98:2 dr, 97% ee

To the solution of 30% H_2O_2 (0.14 mL) in water (0.16 mL) was added $LiOH \cdot H_2O$ (19.2 mg, 0.45 mmol) at 0 °C. The reaction mixture was stirred at 0 °C for 5 min, and the solution of *sulfa*-Michael-Aldol adduct **3a** (90.6 mg, 0.3 mmol) in THF (0.86 mL) was added over 15 min. After completion monitored by TLC then carefully treated with a saturated aqueous solution of $Na_2S_2O_3$ (0.92 mL) followed by 2 M aqueous solution off HCl (2 mL). The mixture was extracted with EtOAc (3 x 5 mL), dried over $MgSO_4$, and removed under reduced pressure to give the crude carboxylic acid product, which can be used without purification.

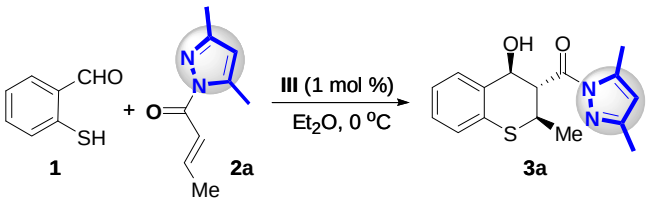
To the solution of the residue in dry DMF (3.0 mL) with stirring, was added CH_3I (64 mg, 0.45 mmol) followed by the addition of Cs_2CO_3 (147 mg, 0.45 mmol) under a positive pressure of argon at room temperature. The resulting mixture was stirred overnight. The mixture was treated with 2 M aqueous solution of HCl, and then extracted with EtOAc (3 x 5 mL), dried over $MgSO_4$, filtered and concentrated in vacuo. The crude product was purified by flash chromatography on silica gel to afford the product **4a** in 75% yield (53.6 mg). $[\alpha]^{25}_D$ +17.6 (*c* 0.3, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 1.30-1.32 (d, *J* = 6.6 Hz, 3H), 2.66-2.73 (m, 1H), 3.60-3.63 (m, 1H), 3.81 (s, 3H), 4.95-4.99 (m, 1H), 7.11-7.15 (m, 3H), 7.60-7.62 (m, 1H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 20.2, 36.6, 52.2, 56.6, 71.0, 124.8, 125.8, 126.1, 127.8, 132.4, 135.8, 173.6; The product was analyzed to determine the diastereoselectivity and enantioselectivity of the reaction: dr = 98:2, 97% ee (major diastereomer) determined by HPLC (Chiralcel AS-H, i-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 254 nm); major diastereomer: t_{minor} = 30.6 min, t_{major} = 13.4 min; minor diastereomer: 10.2 min, 25.1 min.

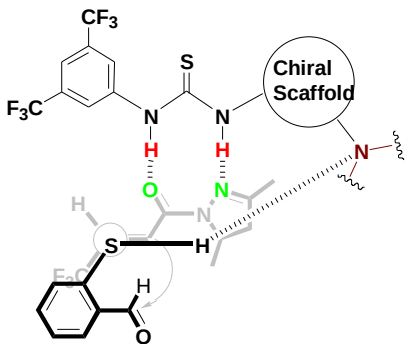
IV. X-ray Crystal Structures of (2*S*,3*R*,4*S*)-**3r**



Crystal data for (2*S*,3*R*,4*S*)-**3r**: $C_{25}H_{22}N_2O_2S$, M_r = 414.51, T = 293 K, Orthorhombic, space group *P*2(1)2(1)2(1), a = 5.8585(10), b = 11.5282(19), c = 30.948(5) Å, V = 2090.2(6) Å^3, Z = 4, 9744 unique reflections, final R_1 = 0.0393 and wR_2 = 0.0910 for 3424 observed [I>2σ(I)] reflections, Flack χ = -0.11(10). CCDC 861217 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/conts/retrieving.html (or from the Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB21EZ, UK; fax: (+44) 1223-336-033; or deposit@ccdc.cam.ac.uk).

V. Proposed Transition-State Model for the Asymmetric Domino *sulfa*-Michael-Aldol Reaction





VI. References

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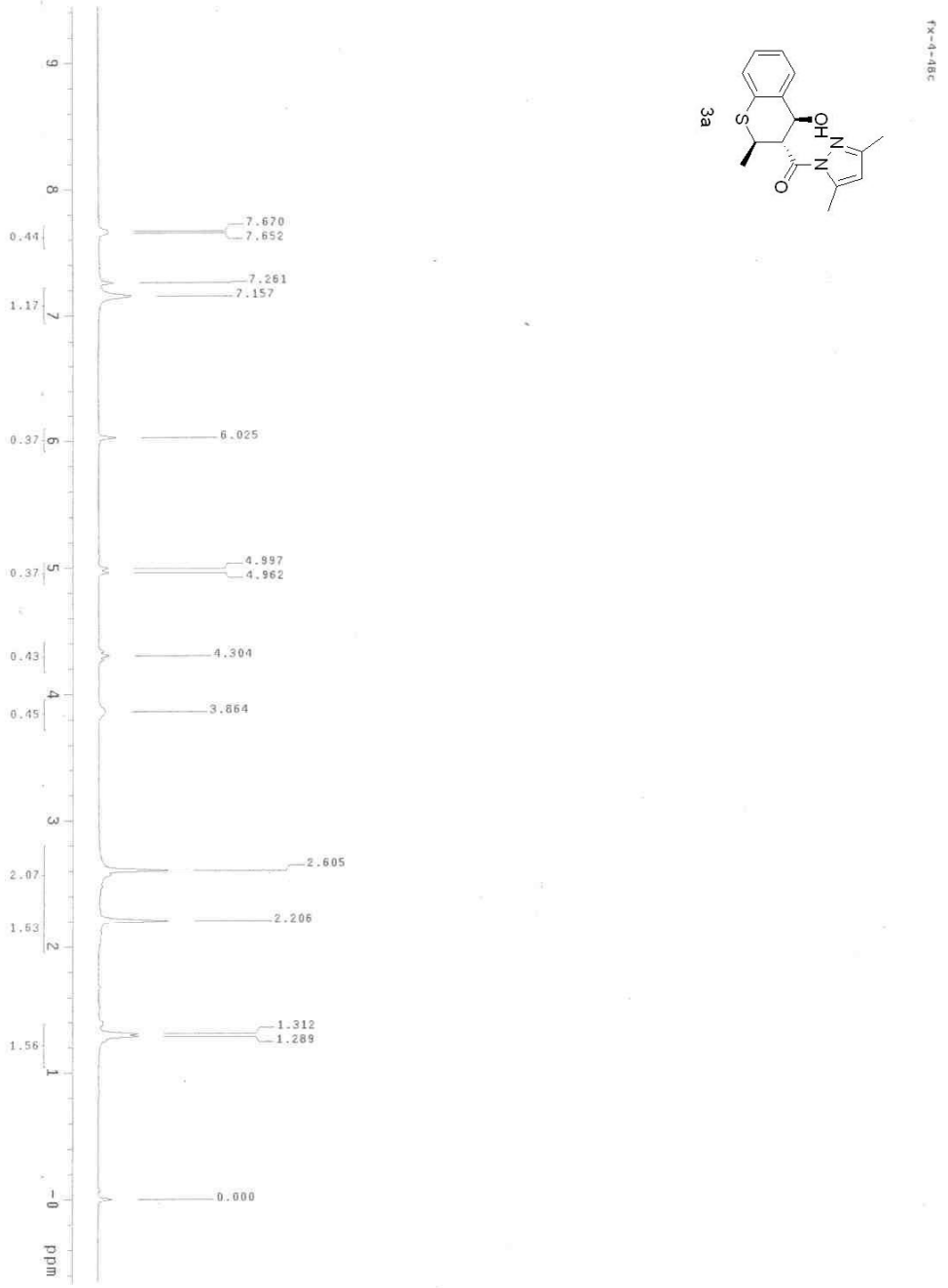
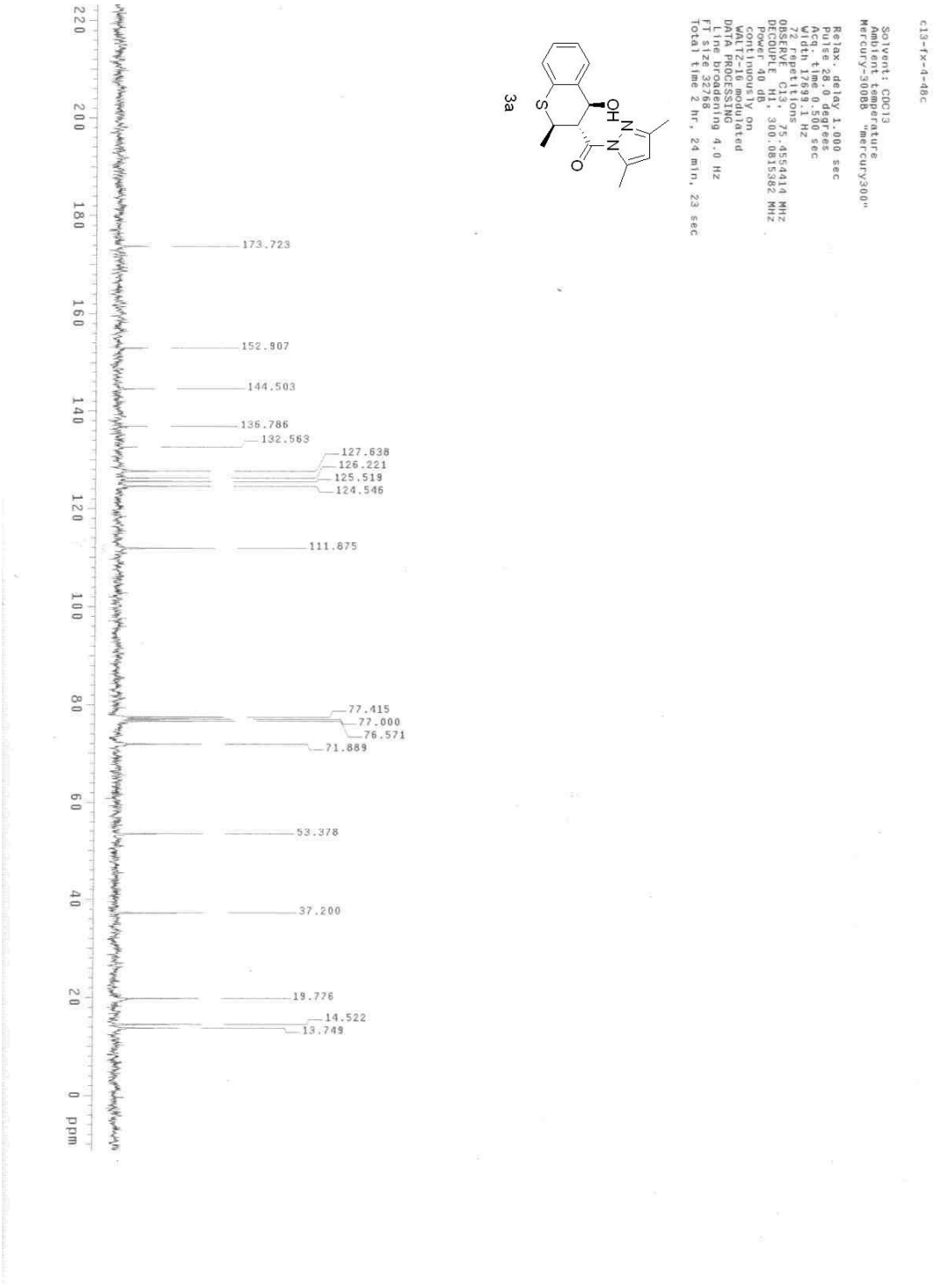
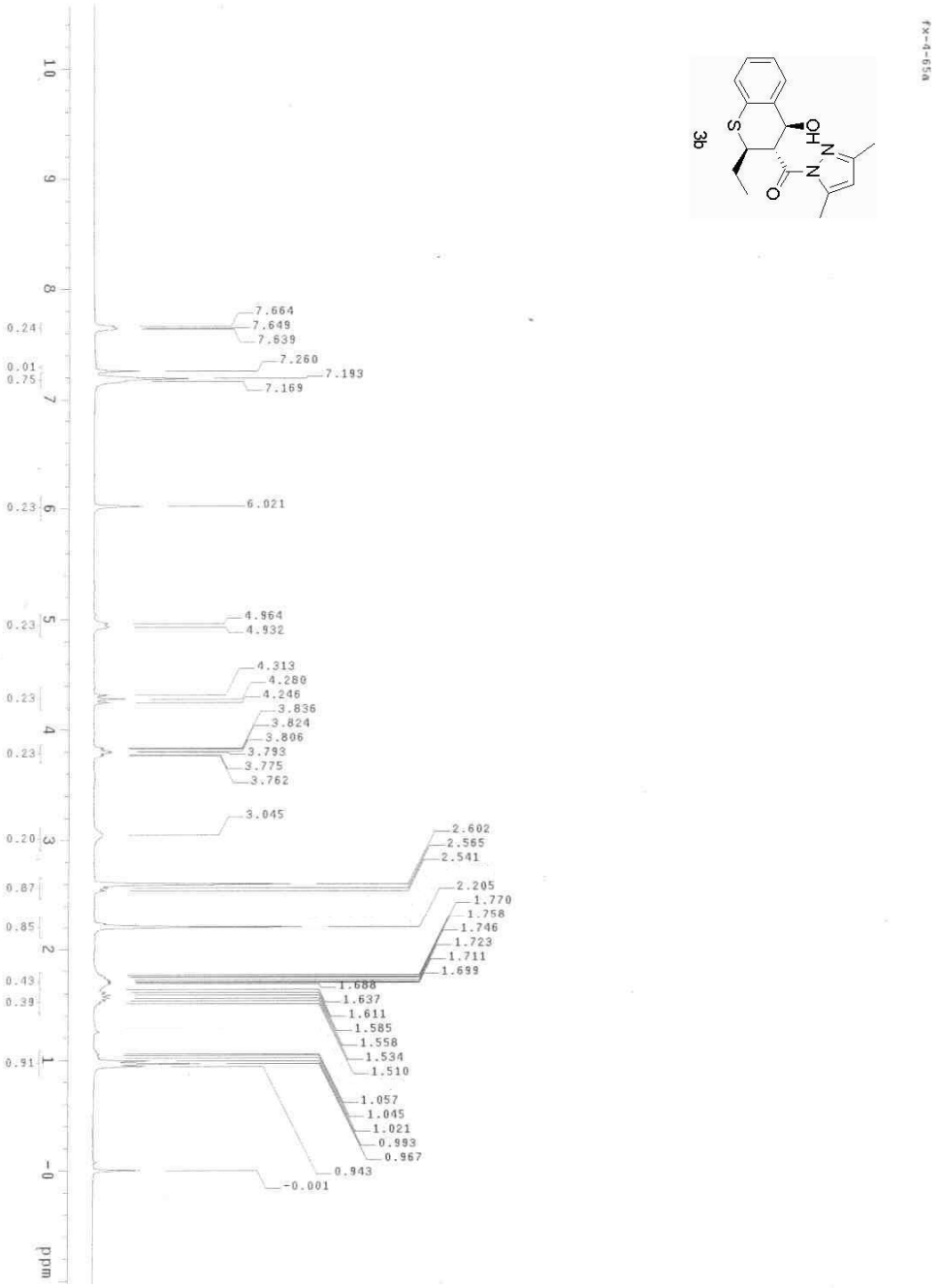
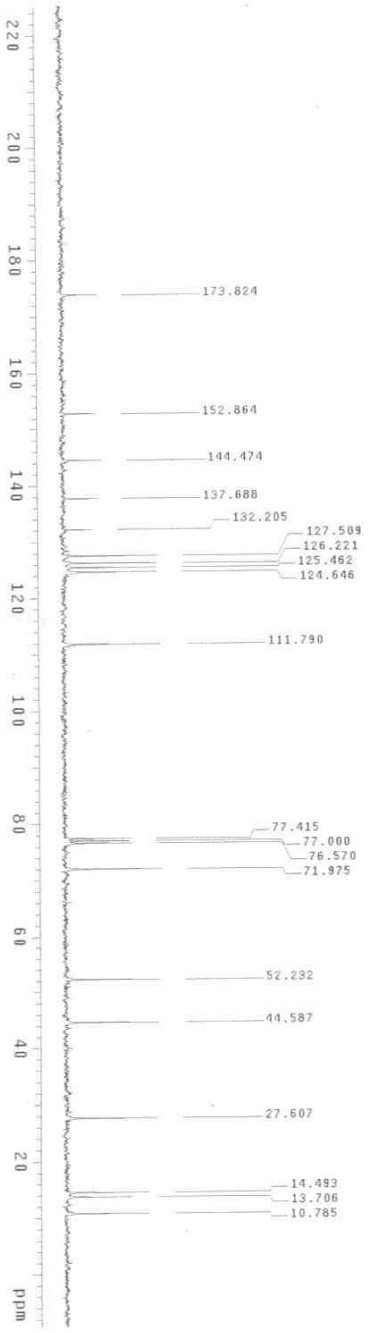
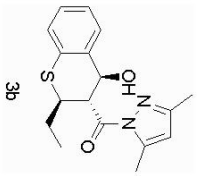
VII. ^{1}H NMR and ^{13}C NMR Spectra




c13-fx-4-48c
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.500 sec
Width 17699.1 Hz
72 repetitions
OBSERVE C13, 75.454414 MHz
DECOUPLE H1, 300.081582 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 2 hr, 24 min, 23 sec
3a
OH
N
N
S
O
173.723
152.907
144.503
136.786
132.563
127.638
126.221
125.519
124.546
111.875
77.415
77.000
76.571
71.889
53.378
37.200
19.776
14.522
13.749
220
200
180
160
140
120
100
80
60
40
20
0
ppm


fx-1-65a
3b
S
OH
N
N
O
7.664
7.649
7.639
7.260
7.193
7.169
6.021
4.964
4.932
4.313
4.280
4.246
3.836
3.824
3.806
3.793
3.775
3.762
3.045
2.602
2.565
2.541
2.205
1.770
1.758
1.746
1.723
1.711
1.699
1.688
1.637
1.611
1.585
1.558
1.534
1.510
1.057
1.045
1.021
0.993
0.967
0.943
-0.001
ppm

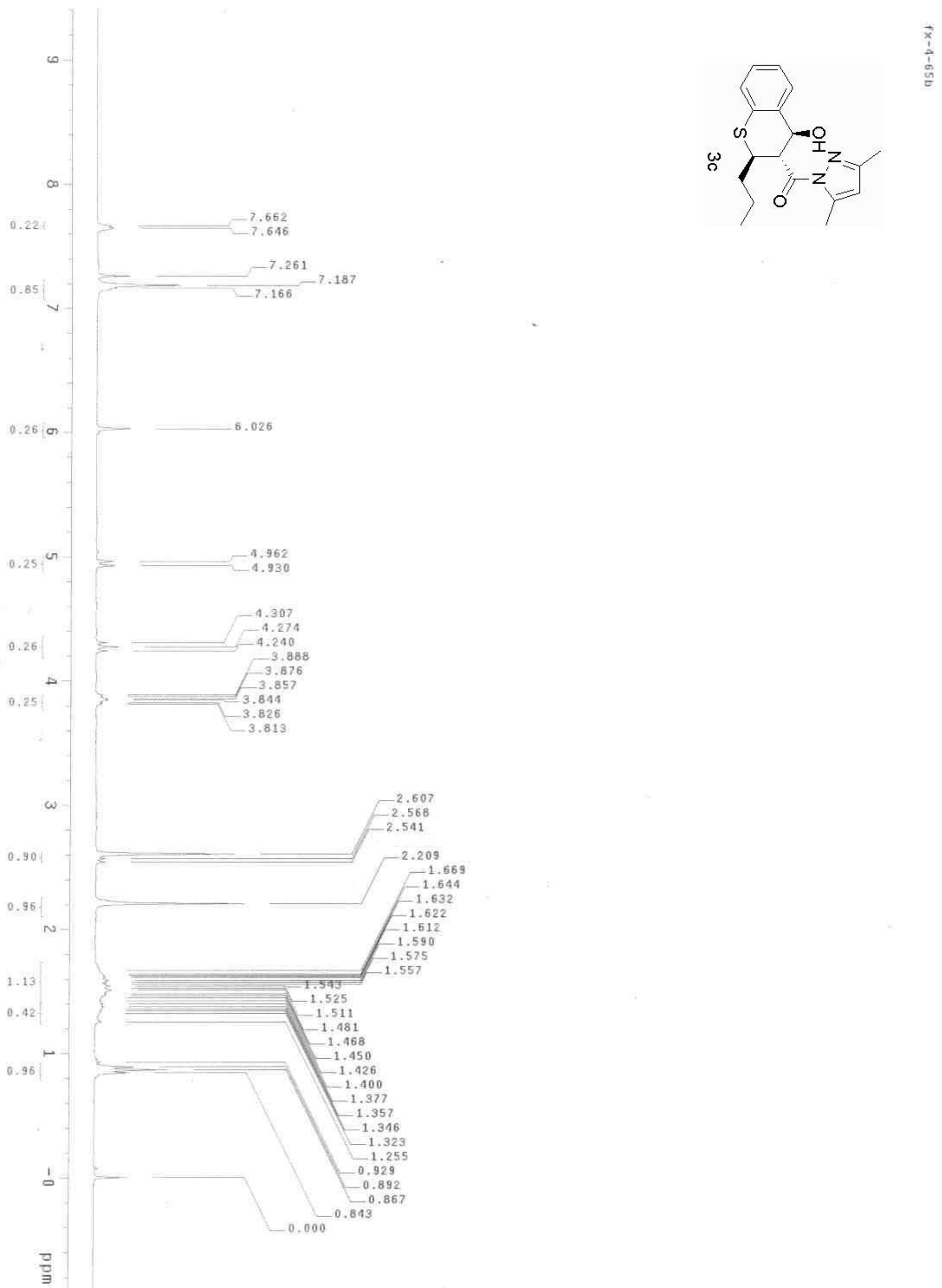


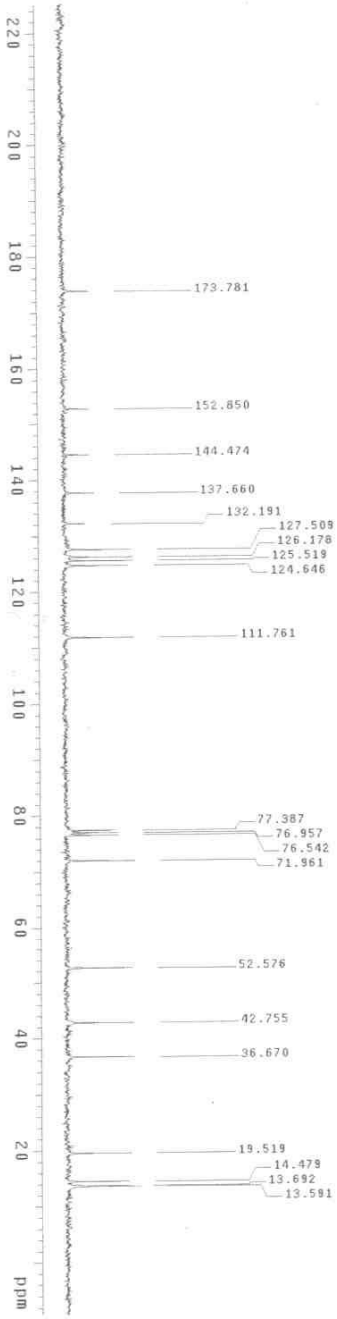


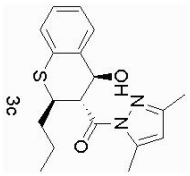
c13fx-4-65a

Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"

Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.500 sec
Width 17699.1 Hz
128 repetitions
OBSERVE C13, 75.4552880 MHz
DECOUPLE H1, 300.0815382 MHz
Power 40 dB
Continuously on
WALTZ16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 2 hr, 24 min, 23 sec


fx-4-65b
3c
S
OH
N
N
O
7.662
7.646
7.261
7.187
7.166
6.026
4.962
4.930
4.307
4.274
4.240
3.888
3.876
3.857
3.844
3.826
3.813
2.607
2.568
2.541
2.209
1.669
1.644
1.632
1.622
1.612
1.590
1.575
1.557
1.543
1.525
1.511
1.481
1.468
1.450
1.426
1.400
1.377
1.357
1.346
1.323
1.255
0.929
0.892
0.867
0.843
0.000
0.22
0.85
0.26
0.25
0.26
0.25
0.90
0.96
1.13
0.42
0.96
ppm

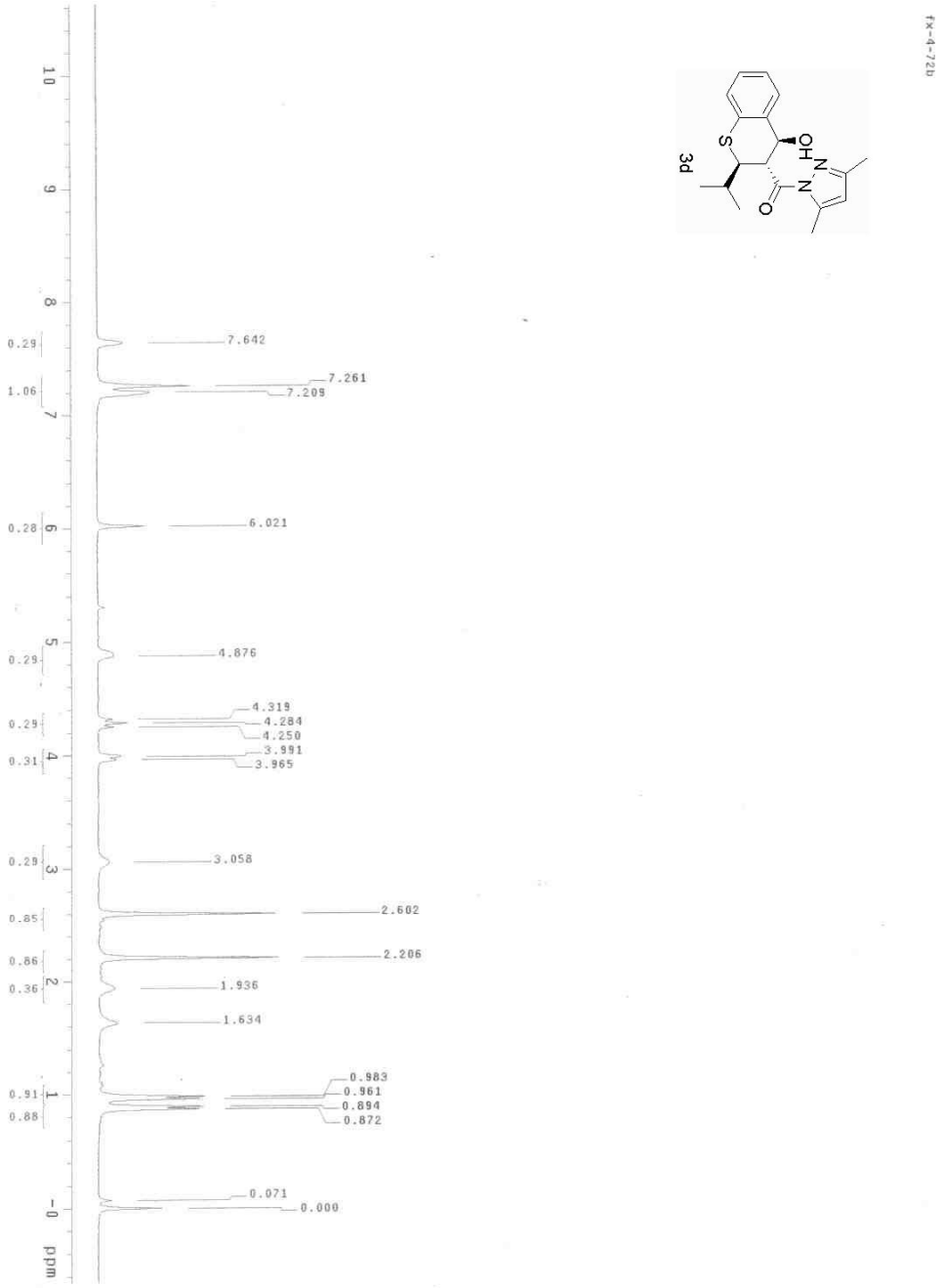


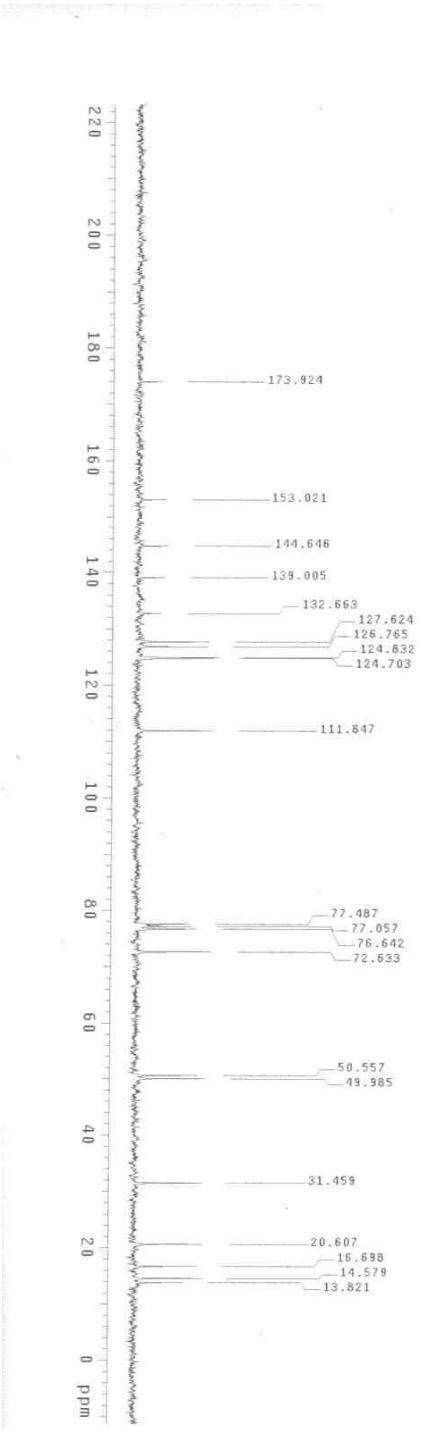


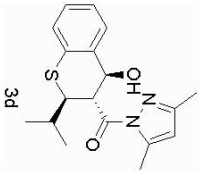
c13fx-4-65b

Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"

Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.500 sec
Width 17699.1 Hz
88 repetitions
OBSERVE C13, 75.4552880 MHz
DECOUPLE H1, 300.0815382 MHz
Power 40 dB
Continuously on
WALTZ16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 2 hr, 24 min, 23 sec


fx-4-72b
3d
S
OH
N
N
O
10
9
8
7
6
5
4
3
2
1
0
ppm
0.29
1.06
0.28
0.29
0.29
0.31
0.29
0.85
0.86
0.36
0.91
0.88
7.642
7.261
7.209
6.021
4.876
4.319
4.284
4.250
3.991
3.965
3.058
2.602
2.206
1.936
1.634
0.983
0.961
0.894
0.872
0.071
0.000

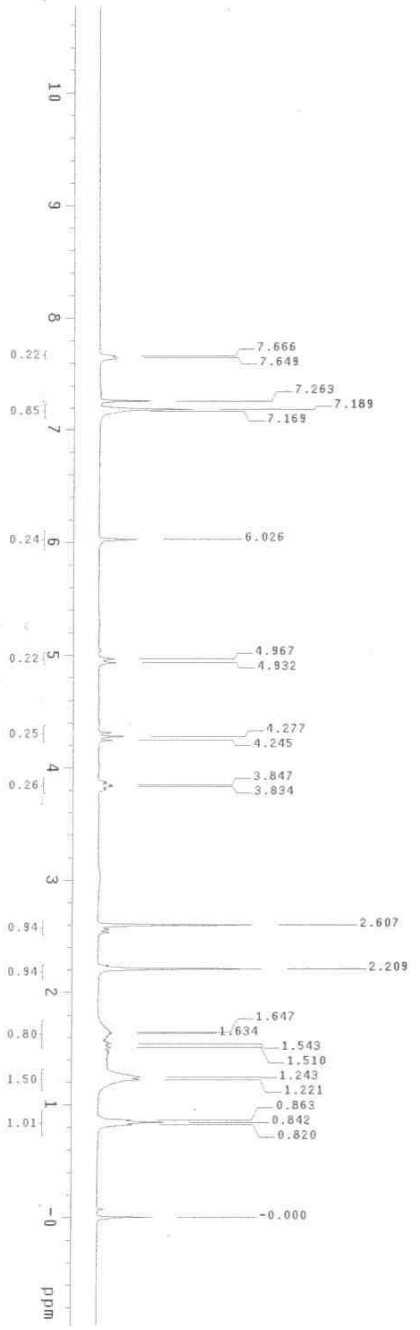


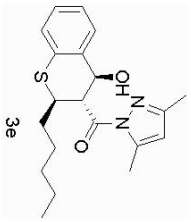


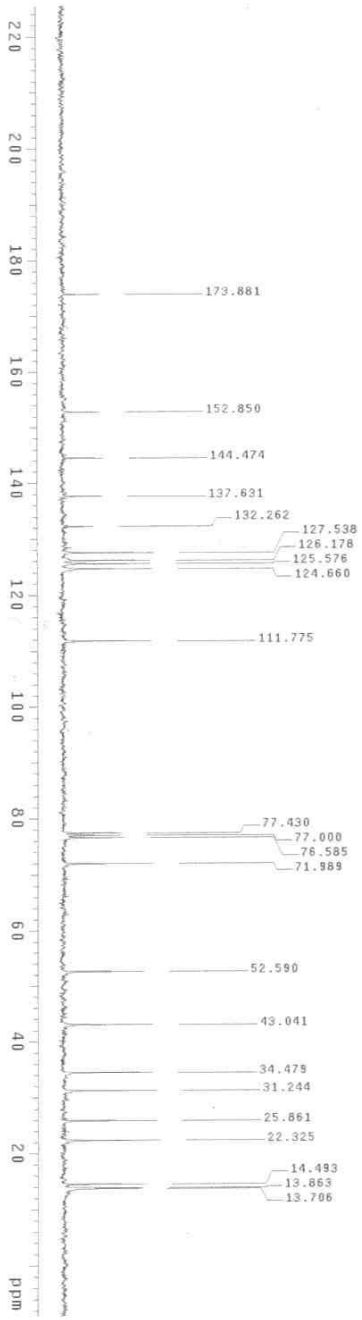
c13-fx-4-72b

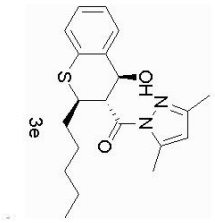
Solvent: CDCl3
Ambient temperature
File: c13-fx-4-72b
Mercury-300BB "mercury300"

Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 1.569 sec
Width 17609.5 Hz
48 repetitions
OBSERVE C13, 75.4554414 MHz
DECOUPLE H1, 300.0815382 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
FT size 32768
Line broadening 4.0 Hz
Total time 2 hr, 24 min, 23 sec


7.666
7.649
7.263
7.189
7.169
6.026
4.967
4.932
4.277
4.245
3.847
3.834
2.607
2.209
1.647
1.634
1.543
1.510
1.243
1.221
0.863
0.842
0.820
-0.000
ppm


3e

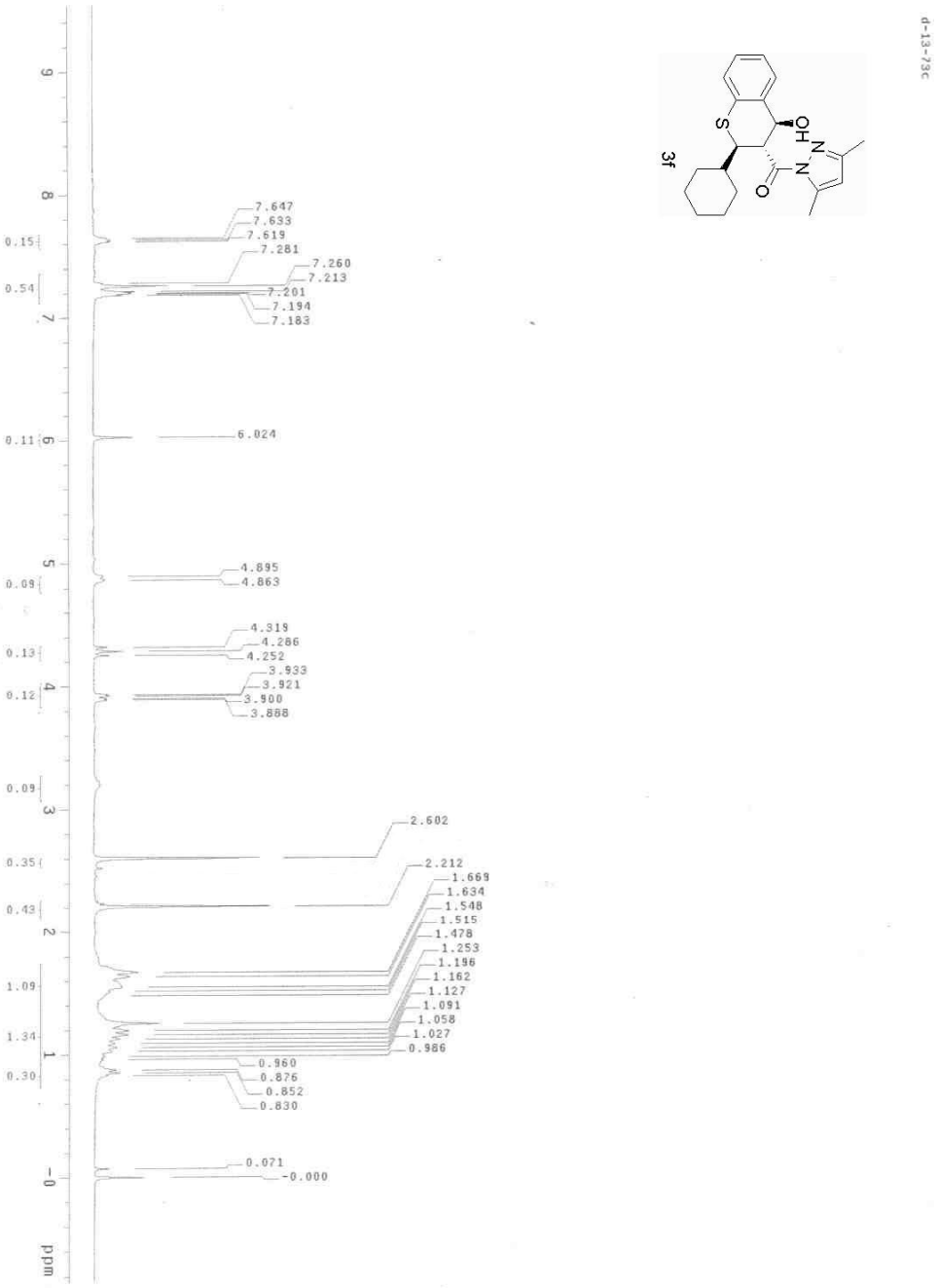


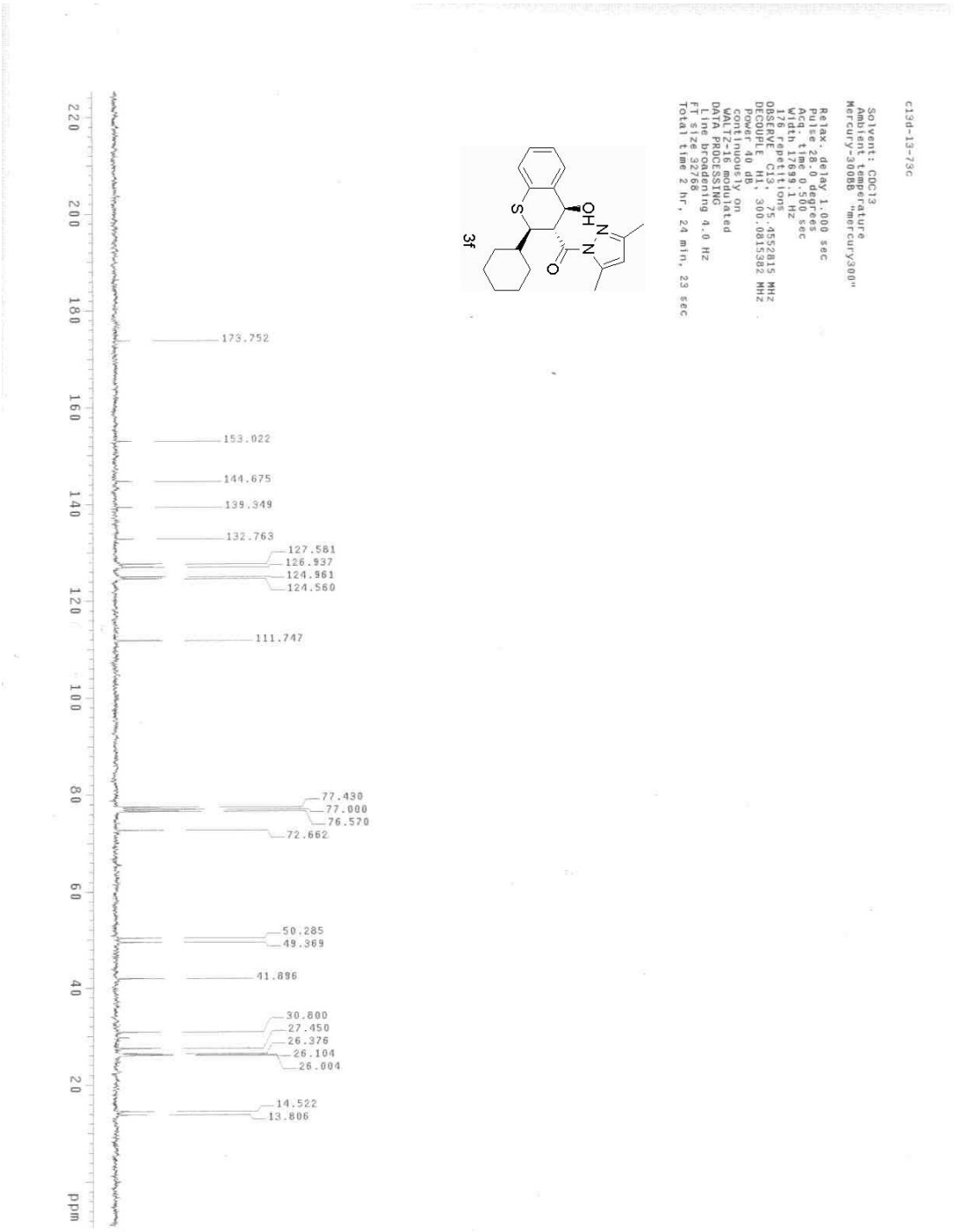


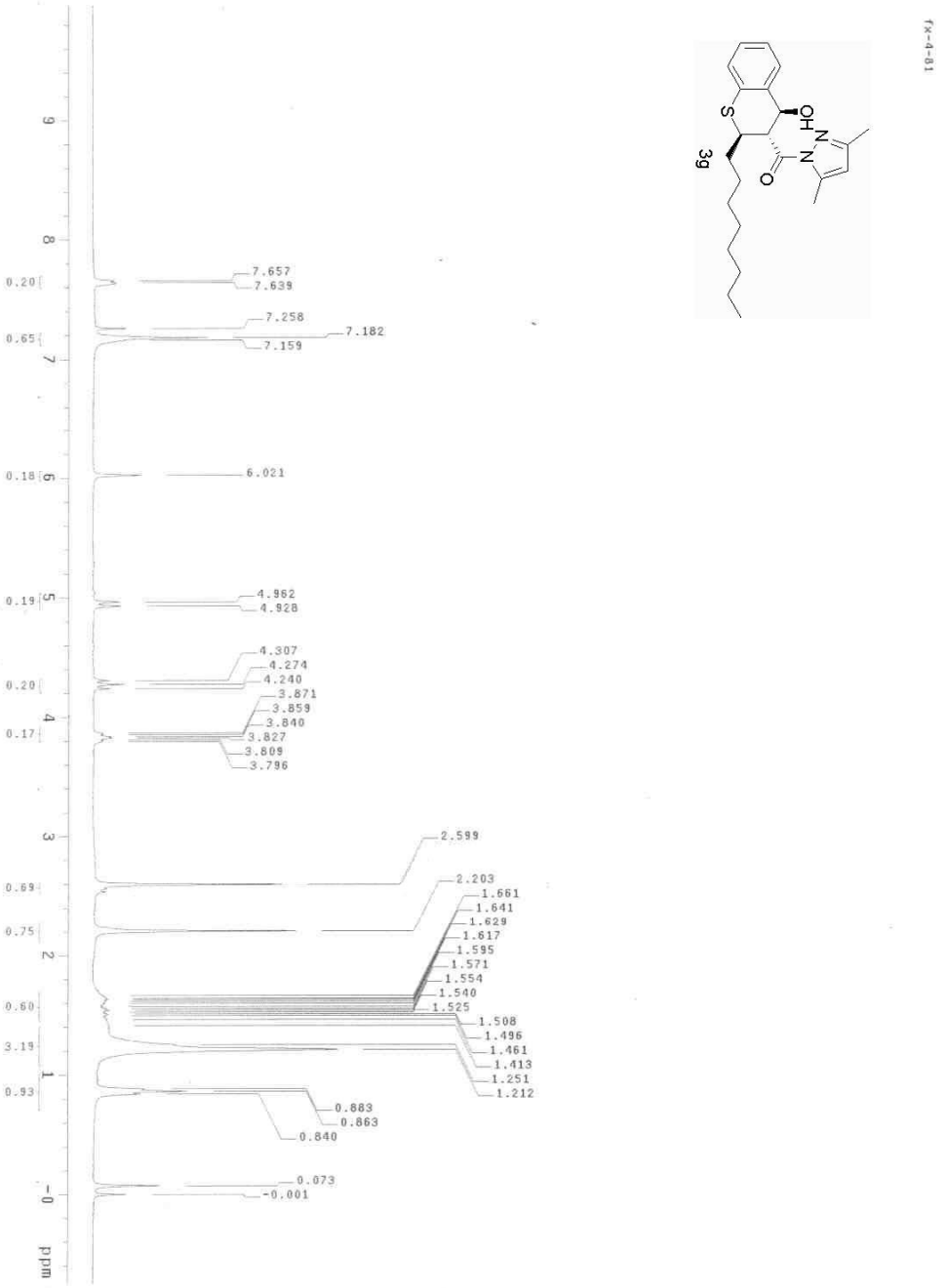
c13fx-4-65c

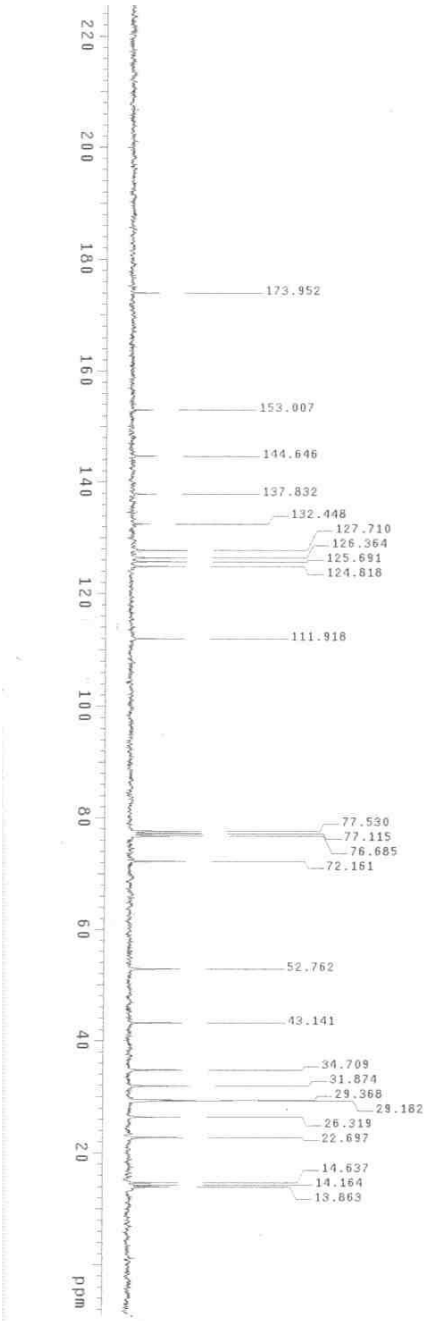
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"

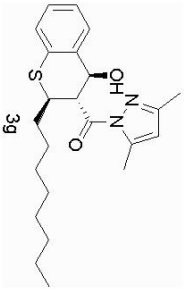
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.500 sec
Width 17699.1 Hz
80 repetitions
OBSERVE C13, 75.4552664 MHz
DECOUPLE H1, 300.0815302 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 2 hr, 24 min, 23 sec


d-13-73c
3f
OH
S
N
N
O
7.647
7.633
7.619
7.281
7.260
7.213
7.201
7.194
7.183
6.024
4.895
4.863
4.319
4.286
4.252
3.933
3.921
3.900
3.888
2.602
2.212
1.669
1.634
1.548
1.515
1.478
1.253
1.196
1.162
1.127
1.091
1.058
1.027
0.986
0.960
0.876
0.852
0.830
0.071
-0.000
0.15
0.54
0.11
0.09
0.13
0.12
0.09
0.35
0.43
1.09
1.34
0.30
ppm


c13d-13-73c
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 0.500 sec
Width 17699.1 Hz
176 repetitions
OBSERVE C13, 75.4552815 MHz
DECOUPLE H1, 300.0815382 MHz
Power 40 dB
continuously on
WALTZ16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 2 hr, 24 min, 23 sec
3f
173.752
153.022
144.675
139.349
132.763
127.581
126.937
124.961
124.560
111.747
77.430
77.000
76.570
72.662
50.285
49.369
41.896
30.800
27.450
26.376
26.104
26.004
14.522
13.806
ppm


fx-4-81
3g
S
OH
N
N
O
9
8
7
6
5
4
3
2
1
0
ppm
0.20
0.65
0.18
0.19
0.20
0.17
0.69
0.75
0.60
3.19
0.93
7.657
7.639
7.258
7.182
7.159
6.021
4.962
4.928
4.307
4.274
4.240
3.871
3.859
3.840
3.827
3.809
3.796
2.599
2.203
1.661
1.641
1.629
1.617
1.595
1.571
1.554
1.540
1.525
1.508
1.496
1.461
1.413
1.251
1.212
0.883
0.863
0.840
0.073
-0.001

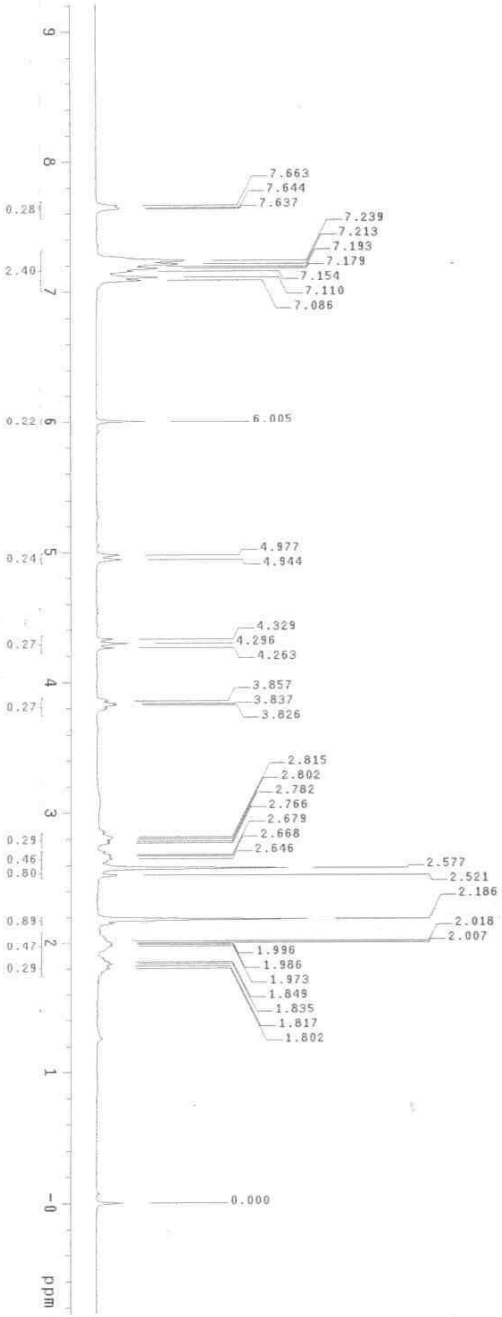


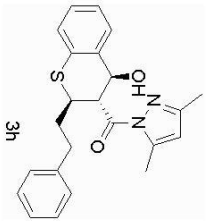


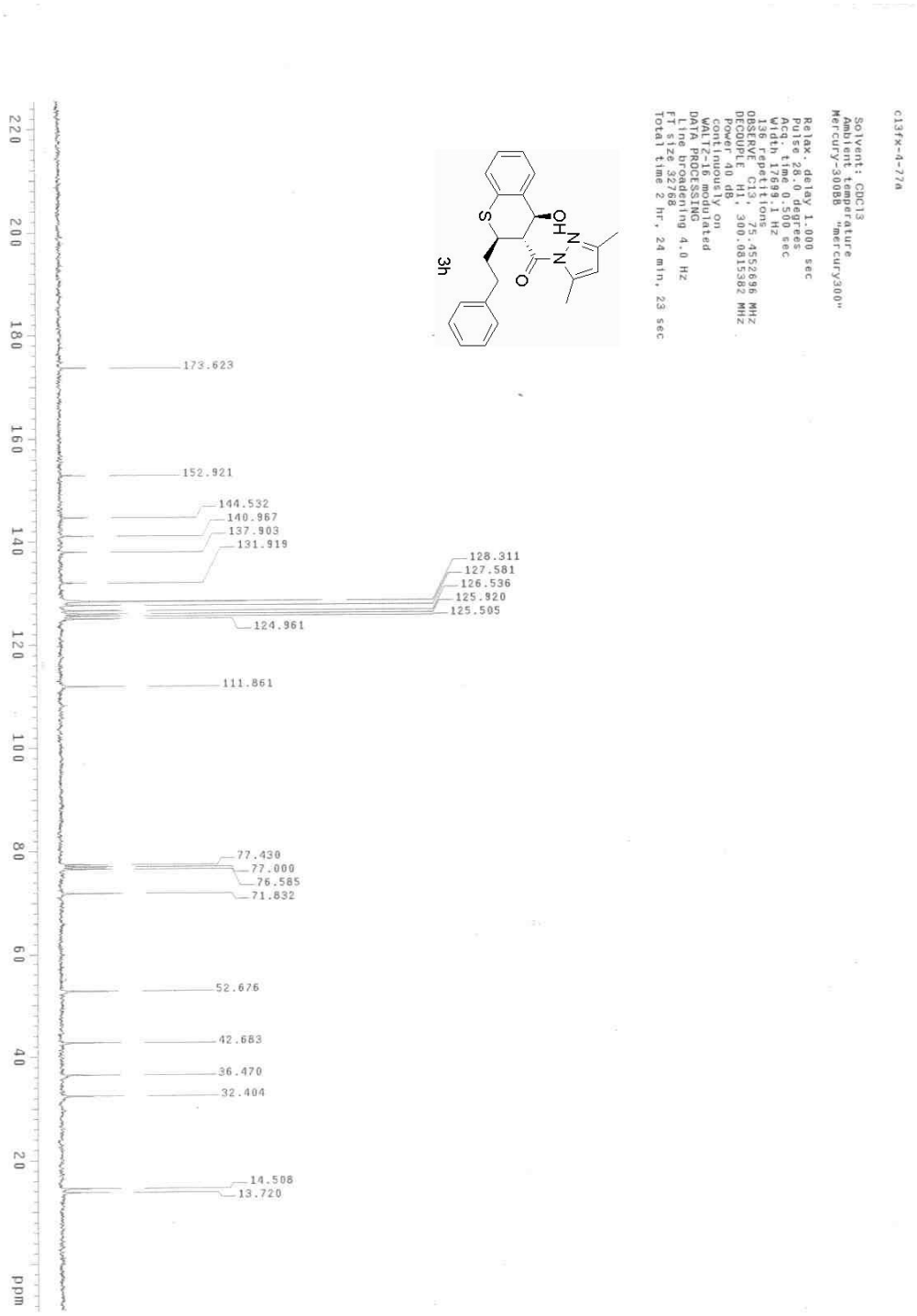
c13fx-4-81

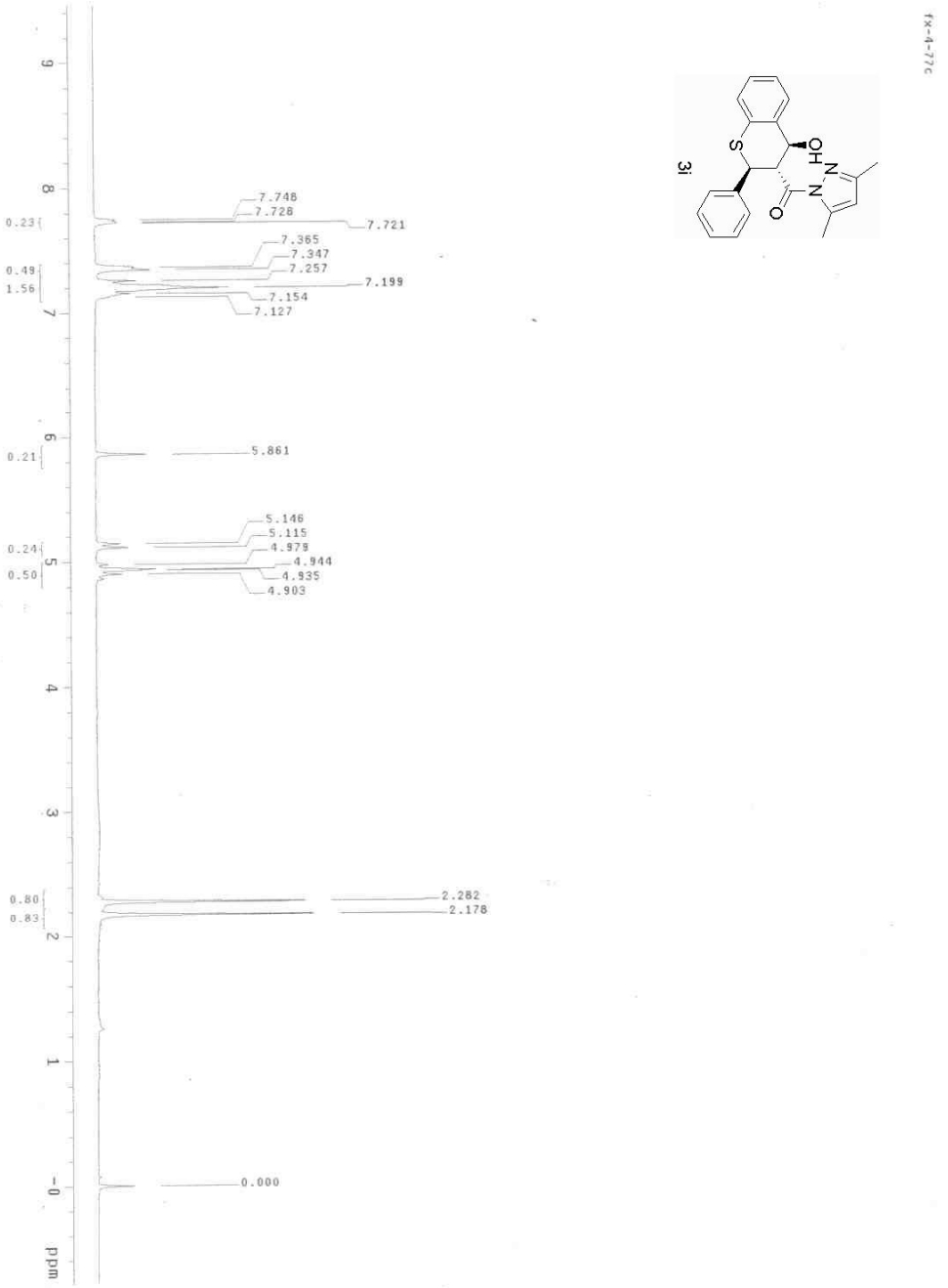
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"

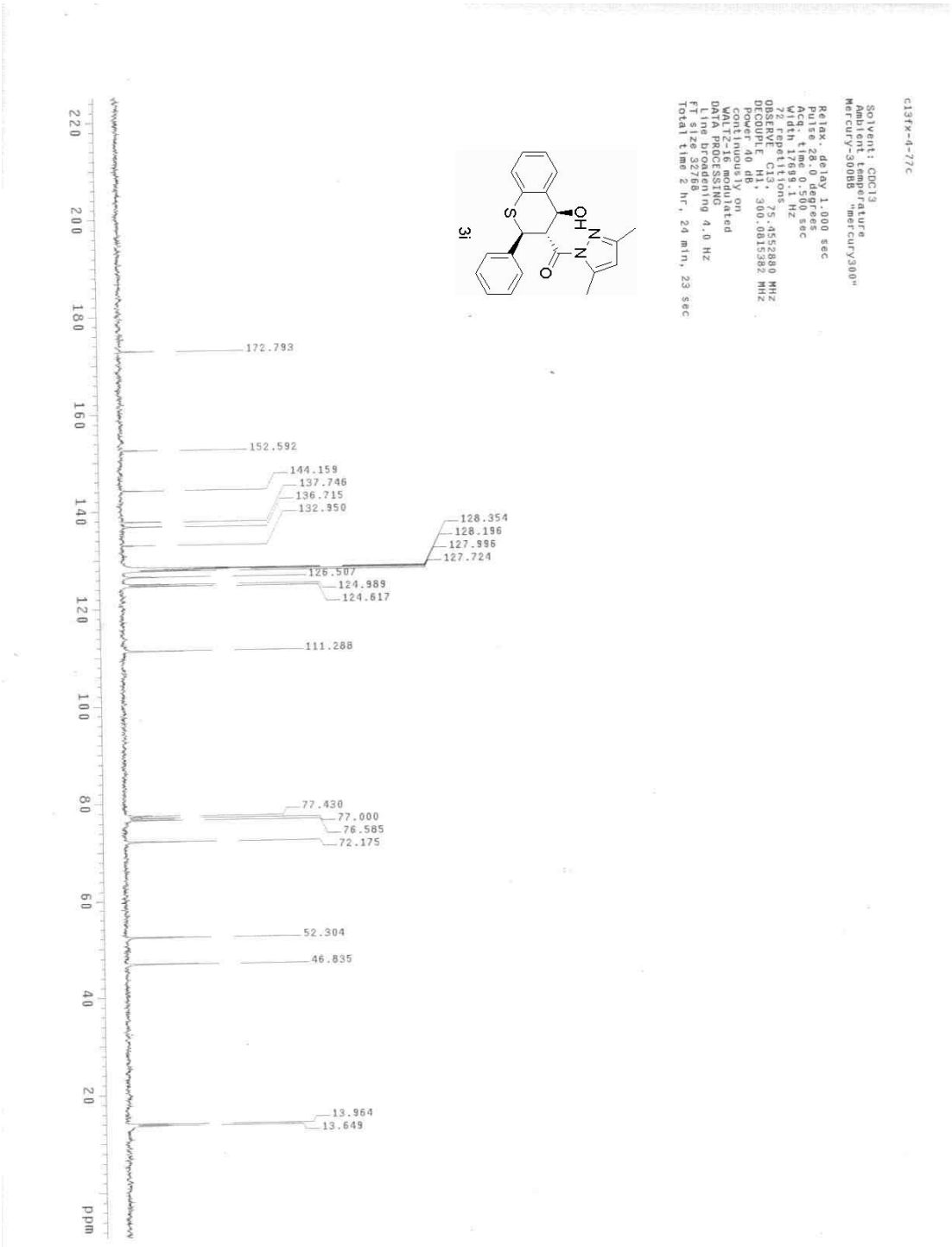
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.500 sec
Width 17699.1 Hz
232 repetitions
OBSERVE C13, 75.455264 MHz
DECOUPLE H1, 300.081582 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 2 hr, 24 min, 23 sec

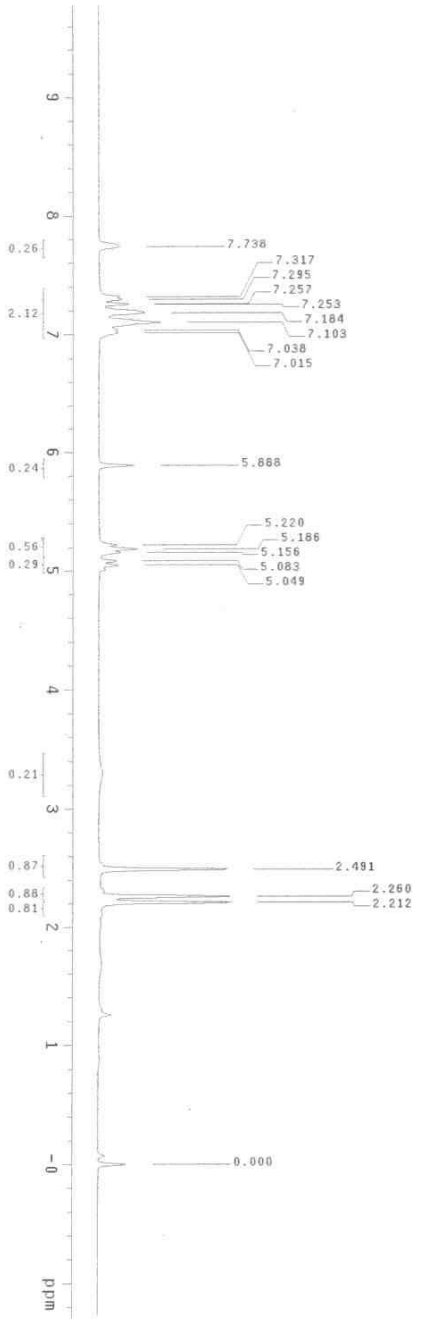

7.663
7.644
7.637
7.239
7.213
7.193
7.179
7.154
7.110
7.086
6.005
4.977
4.944
4.329
4.296
4.263
3.857
3.837
3.826
2.815
2.802
2.782
2.766
2.679
2.668
2.646
2.577
2.521
2.186
2.018
2.007
1.996
1.986
1.973
1.849
1.835
1.817
1.802
0.000
ppm

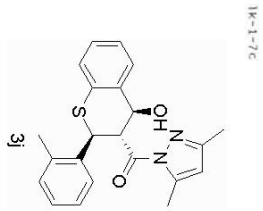

3h

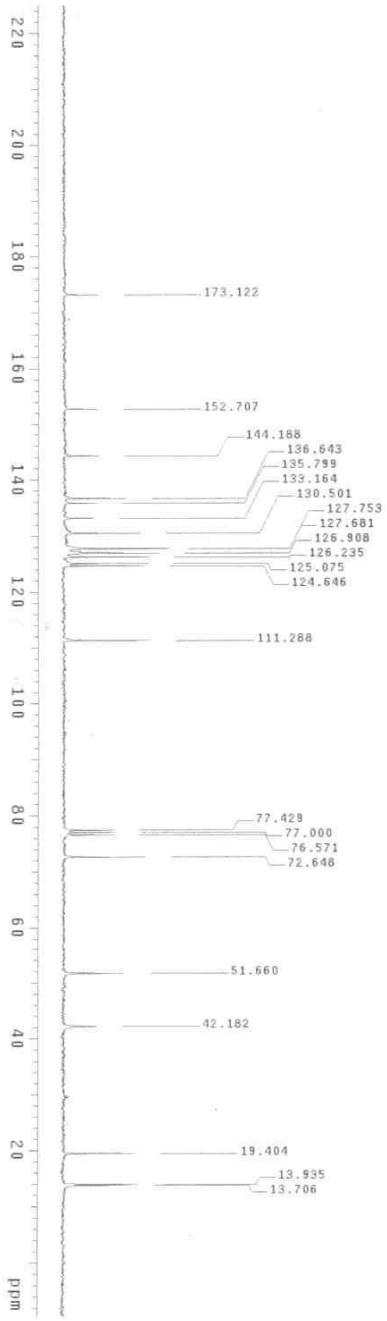

c13fx-4-77a
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.500 sec
Width 17699.1 Hz
136 repetitions
OBSERVE C13, 75.455696 MHz
DECOUPLE H1, 300.0815382 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 2 hr, 24 min, 23 sec
3h
173.623
152.921
144.532
140.967
137.903
131.919
128.311
127.581
126.536
125.920
125.505
124.961
111.861
77.430
77.000
76.585
71.832
52.676
42.683
36.470
32.404
14.508
13.720
ppm

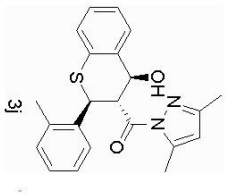

fx-4-77c
3i
S
OH
N
N
O
9
8
7
6
5
4
3
2
1
0
ppm
0.23
0.49
1.56
0.21
0.24
0.50
0.80
0.83
7.748
7.728
7.721
7.365
7.347
7.257
7.199
7.154
7.127
5.861
5.146
5.115
4.979
4.944
4.935
4.903
2.282
2.178
0.000


c13fx-4-77c
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.500 sec
Width 17699.1 Hz
72 repetitions
OBSERVE C13, 75.455280 MHz
DECOUPLE H1, 300.081582 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 2 hr, 24 min, 23 sec
3i
OH
N
N
S
O
172.793
152.592
144.159
137.746
136.715
132.950
128.354
128.196
127.996
127.724
126.507
124.989
124.617
111.288
77.430
77.000
76.585
72.175
52.304
46.835
13.964
13.649
220
200
180
160
140
120
100
80
60
40
20
ppm


7.738
7.317
7.295
7.257
7.253
7.184
7.103
7.038
7.015
5.888
5.220
5.186
5.156
5.083
5.049
2.491
2.260
2.212
0.000
0.26
2.12
0.24
0.56
0.29
0.21
0.87
0.86
0.81
ppm


3j
S
OH
N
N
O

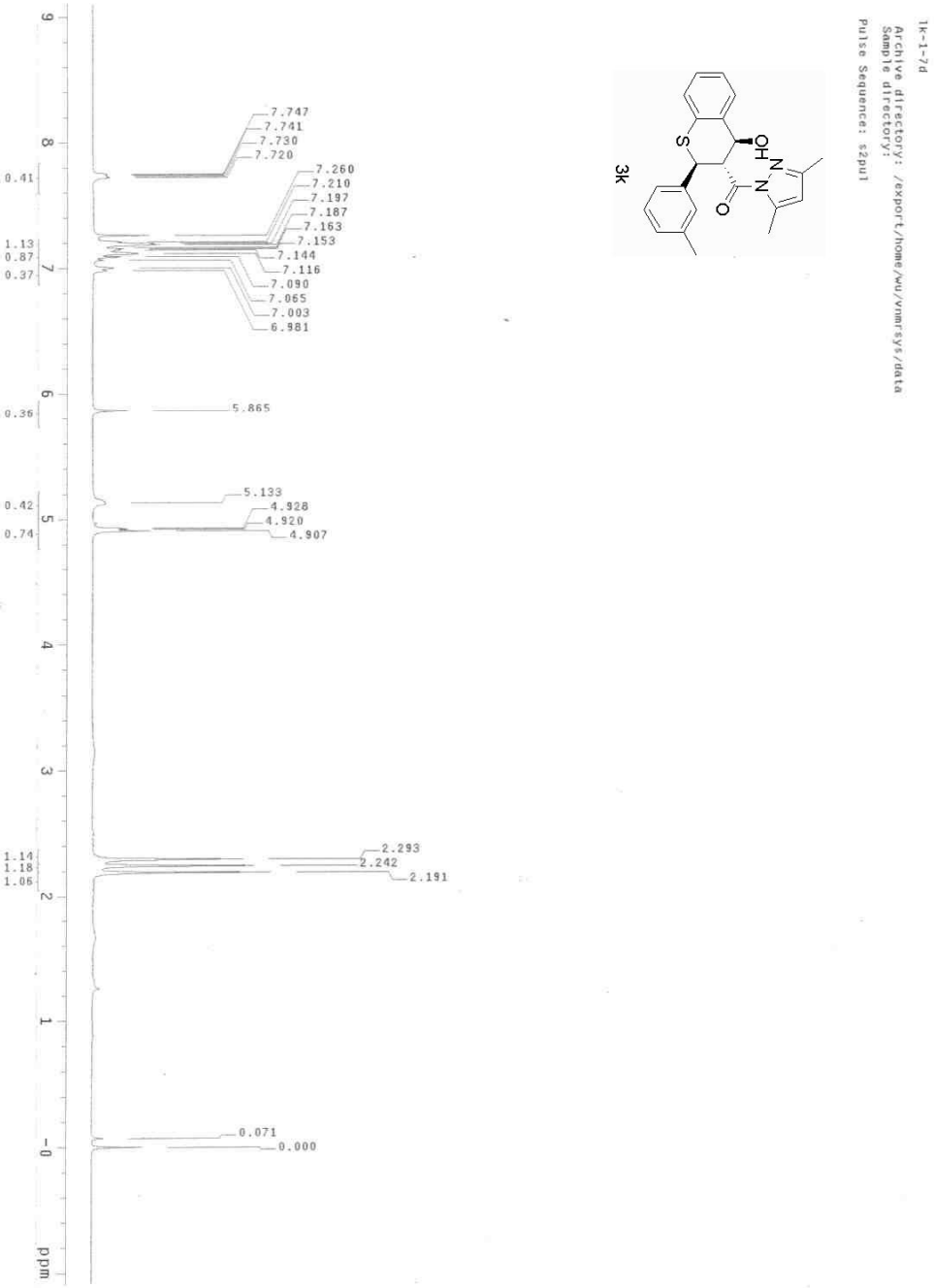


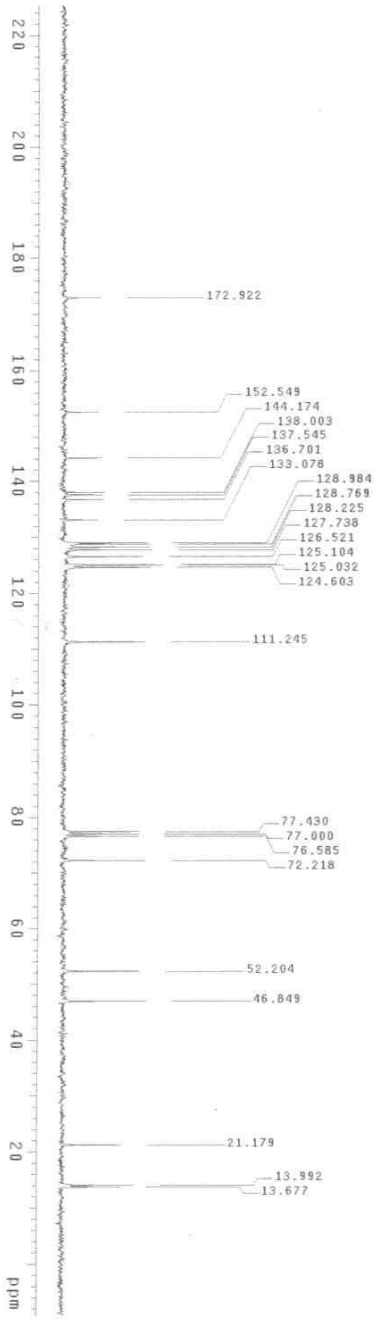


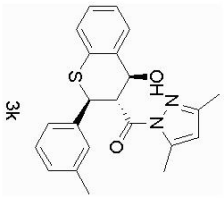
c131k-1-7c

Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"

Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.500 sec
Width 17699.1 Hz
224 repetitions
OBSERVE C13, 75.4553107 MHz
DECOUPLE H1, 300.0815382 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 2 hr, 24 min, 23 sec


1k-1-7d
Archive directory: /export/home/wu/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
3k
7.747
7.741
7.730
7.720
7.260
7.210
7.197
7.187
7.163
7.153
7.144
7.116
7.090
7.065
7.003
6.981
5.865
5.133
4.928
4.920
4.907
2.293
2.242
2.191
0.071
0.000
0.41
1.13
0.87
0.37
0.36
0.42
0.74
1.14
1.18
1.06
ppm

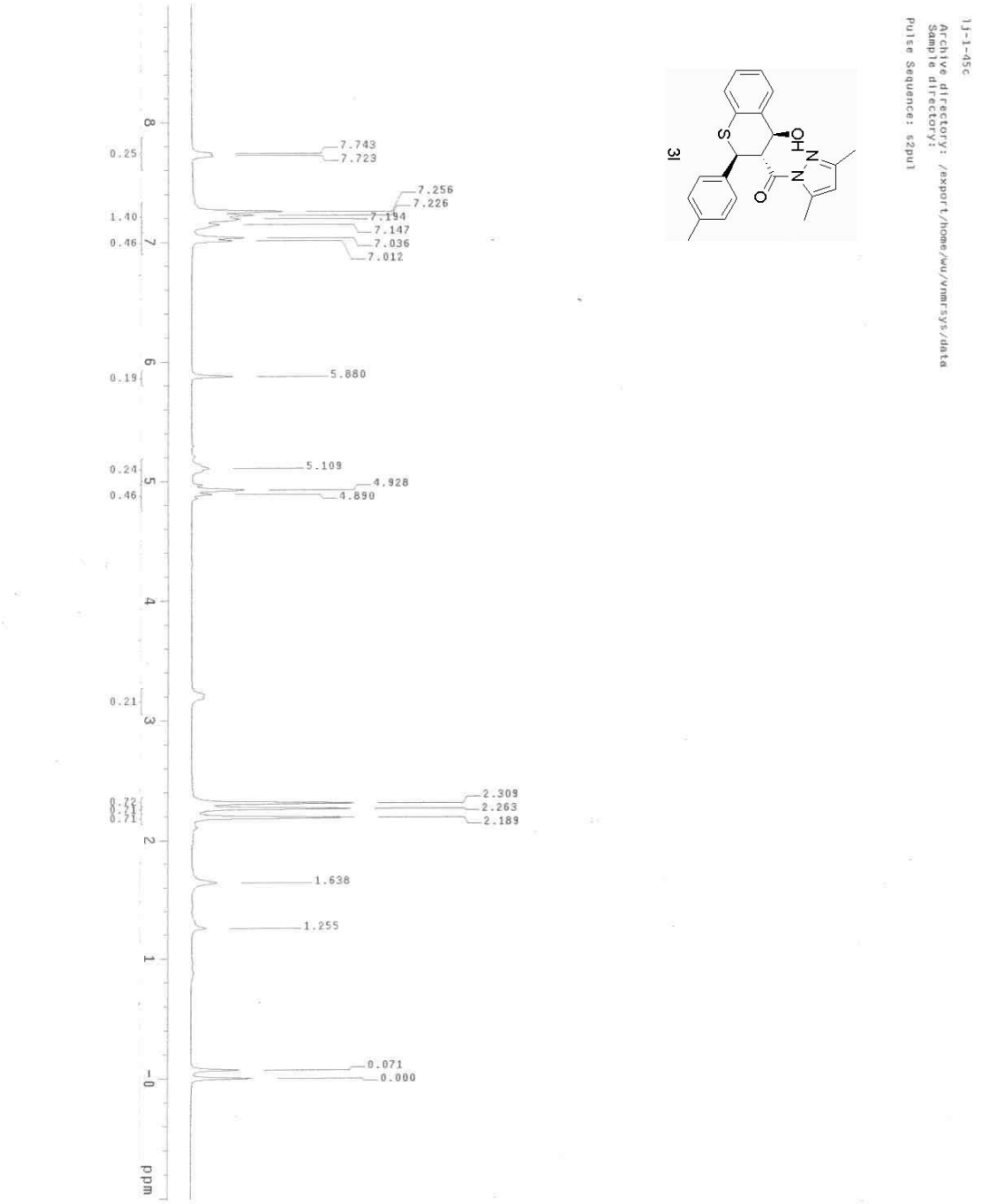


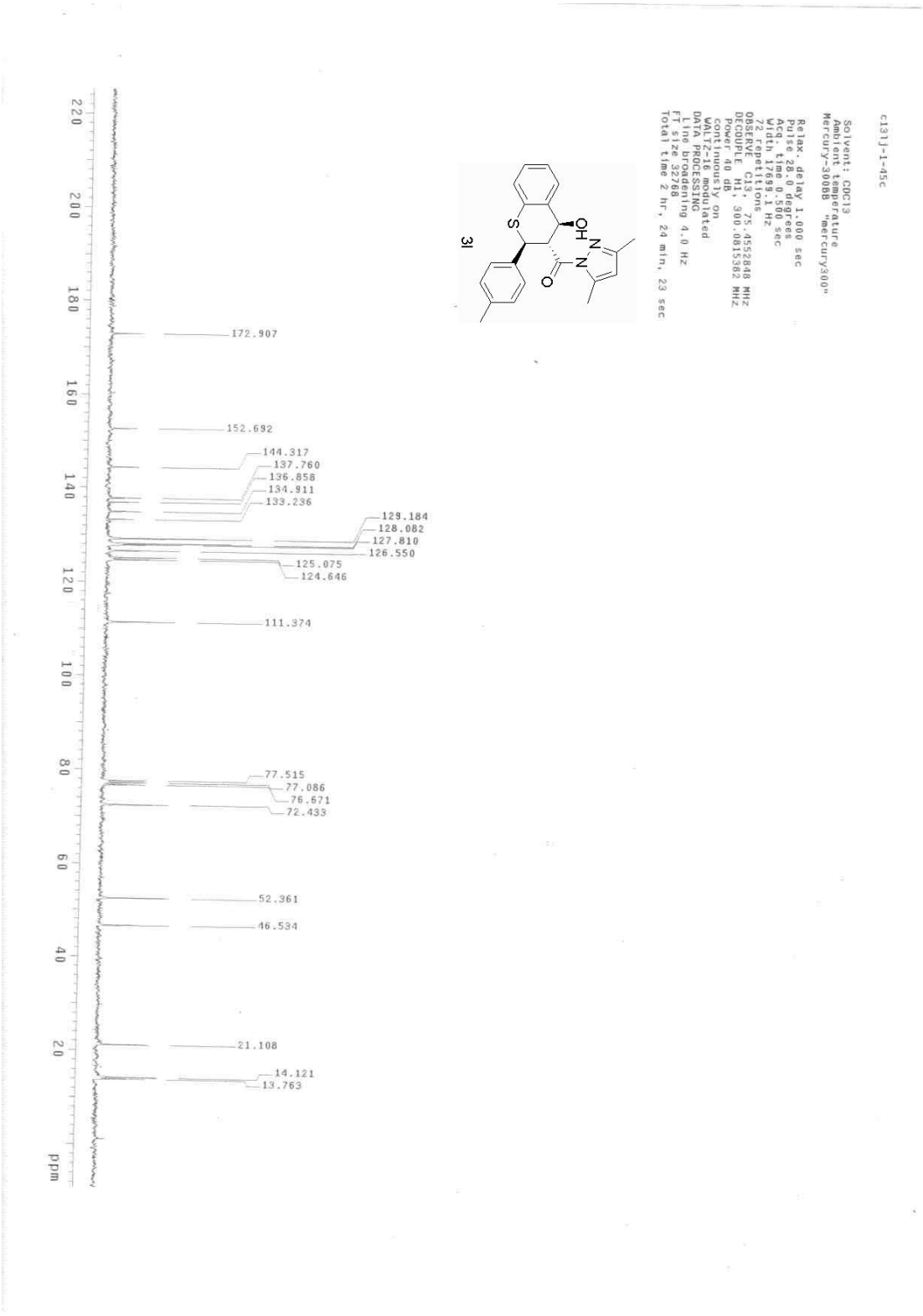


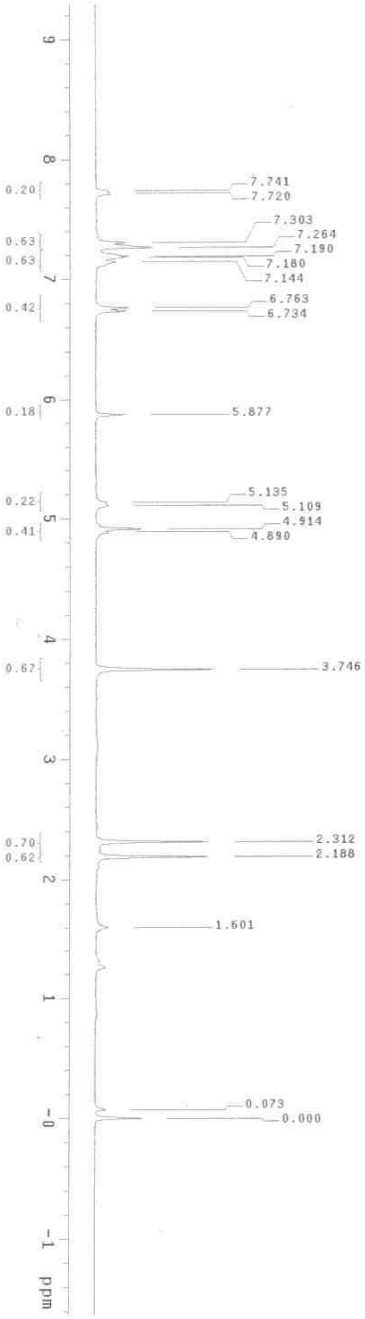
c13lk-1-7d

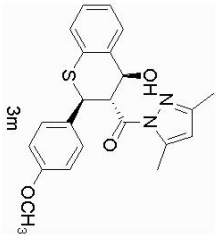
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"

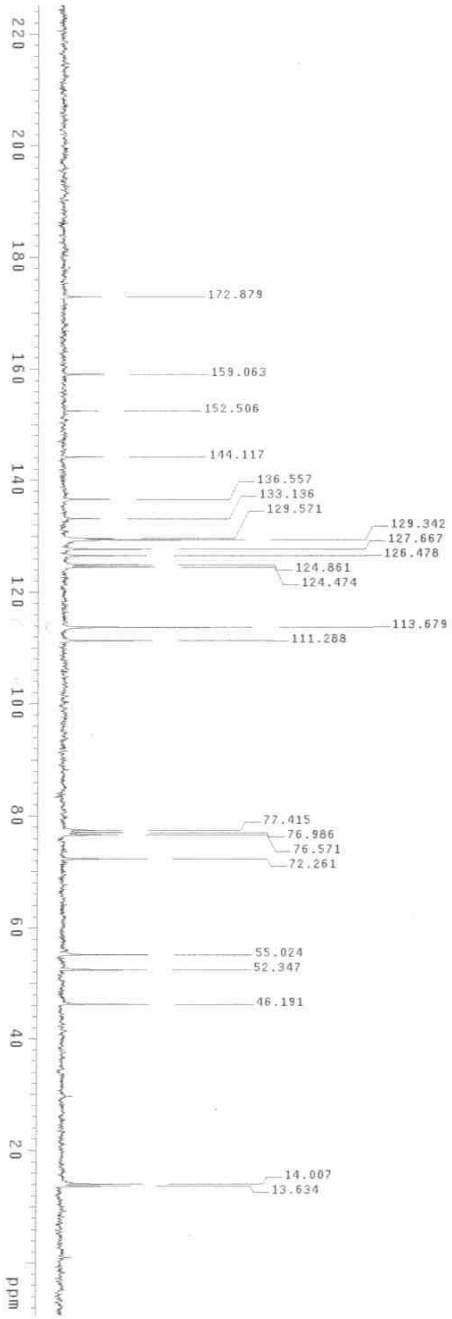
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.500 sec
Width 17699.1 Hz
80 repetitions
OBSERVE C13, 75.455848 MHz
DECOUPLE H1, 300.081582 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 2 hr, 24 min, 23 sec

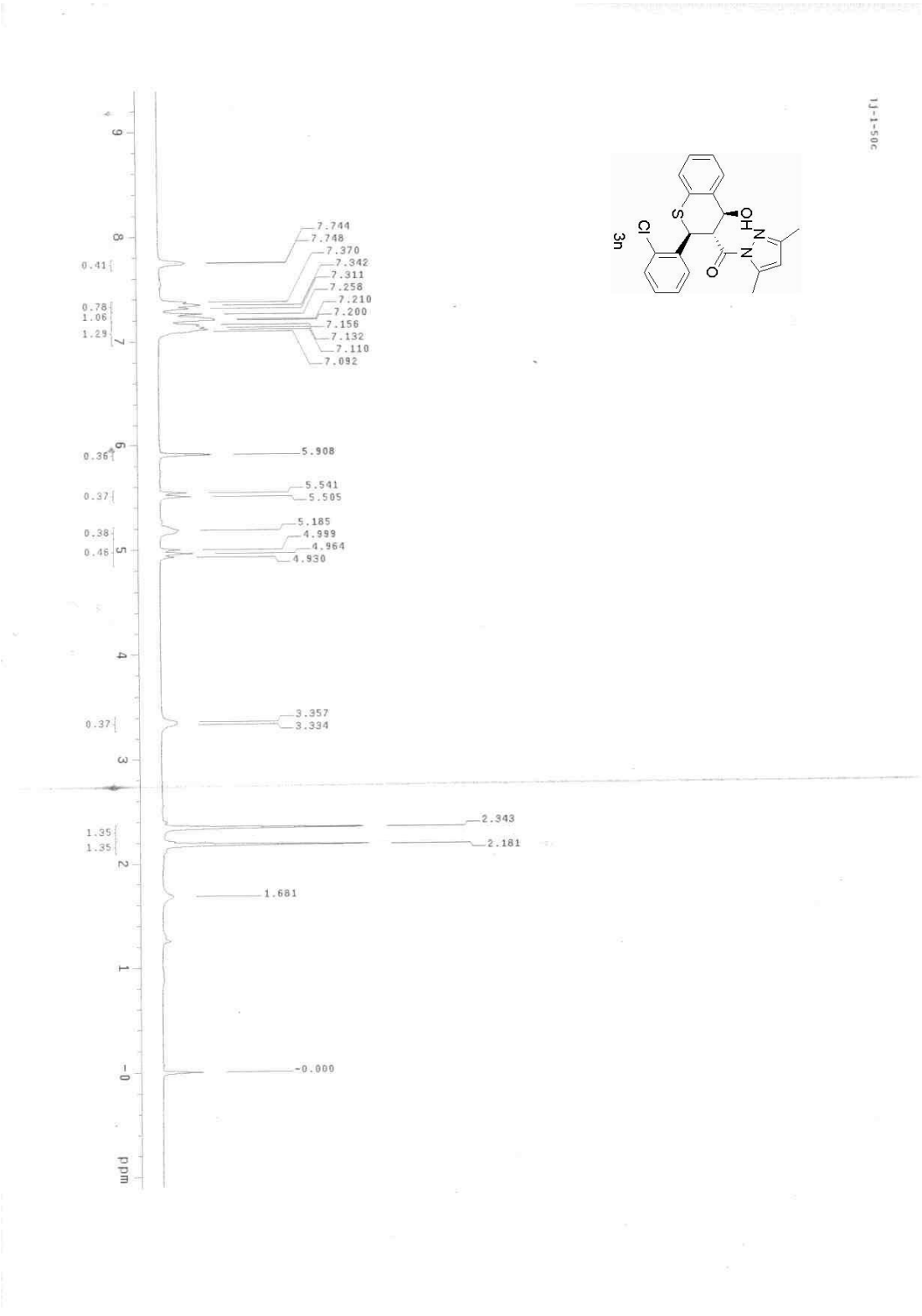

lj-1-45c
Archive directory: /export/home/wu/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
3l
7.743
7.723
7.256
7.226
7.194
7.147
7.036
7.012
5.880
5.109
4.928
4.890
2.309
2.263
2.189
1.638
1.255
0.071
0.000
ppm

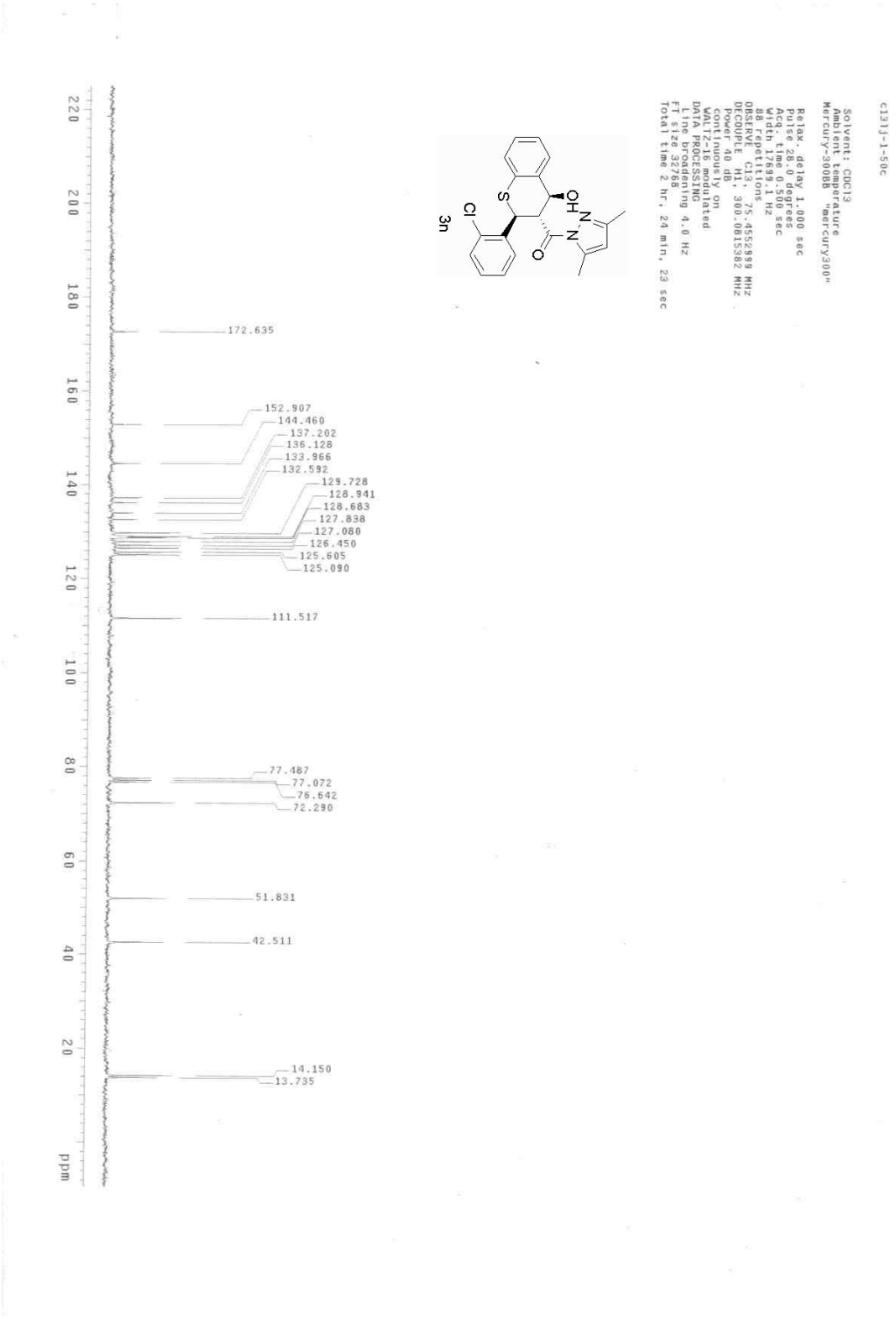

c131j-1-45c
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.500 sec
Width 17699.1 Hz
72 repetitions
OBSERVE C13, 75.4552848 MHz
DECOUPLE H1, 300.0815302 MHz
Power 60 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 2 hr, 24 min, 23 sec
3l
S
OH
N
N
O
172.907
152.692
144.317
137.760
136.858
134.911
133.236
129.184
128.082
127.810
126.550
125.075
124.646
111.374
77.515
77.086
76.671
72.433
52.361
46.534
21.108
14.121
13.763
220
200
180
160
140
120
100
80
60
40
20
ppm

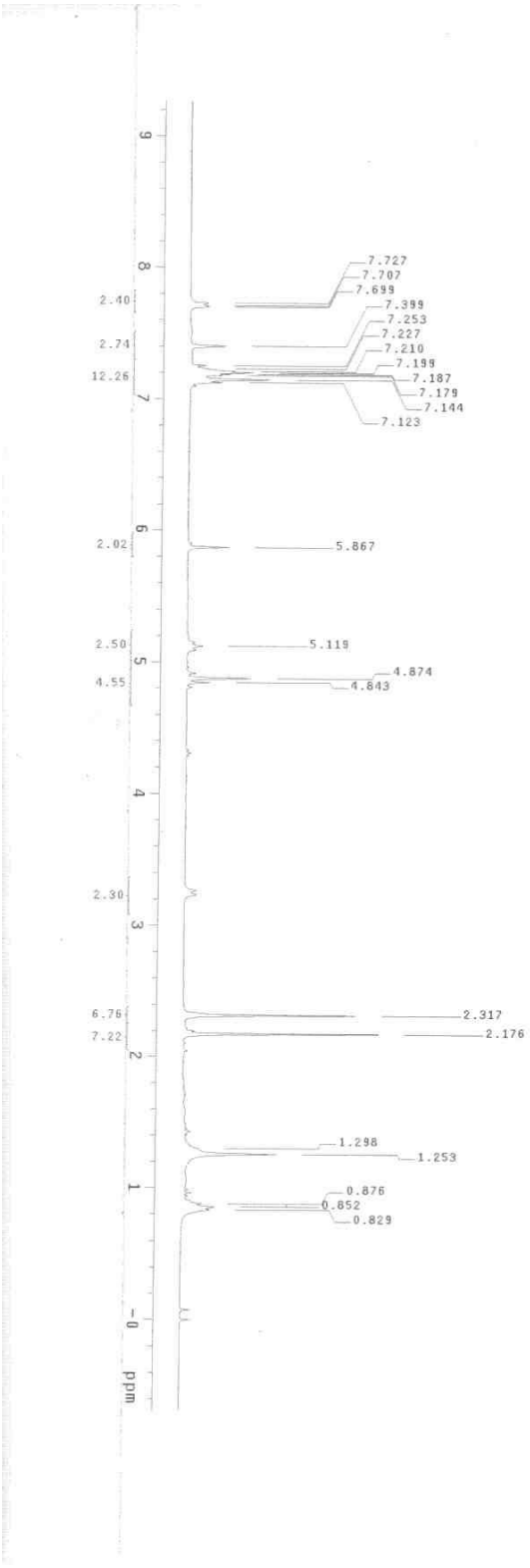

7.741
7.720
7.303
7.264
7.190
7.180
7.144
6.763
6.734
5.877
5.135
5.109
4.914
4.890
3.746
2.312
2.188
1.601
0.073
0.000
0.20
0.63
0.63
0.42
0.18
0.22
0.41
0.67
0.70
0.62
ppm

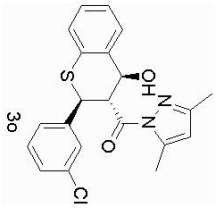

3m
OCH3
OH
N
N
O
S

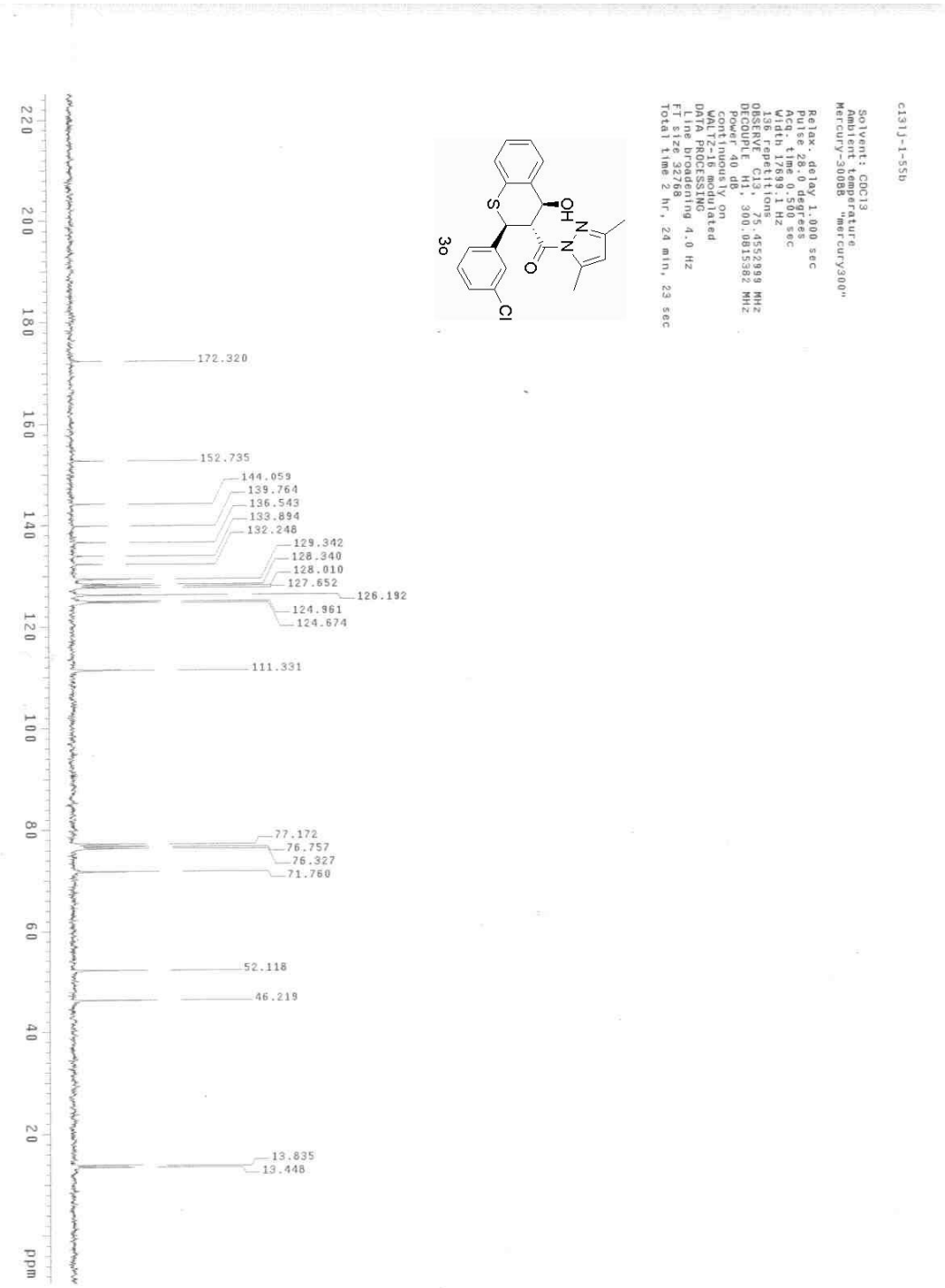

c131j-1-45d
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.500 sec
Width 17699.1 Hz
48 repetitions
OBSERVE C13, 75.455999 MHz
DECOUPLE H1, 300.081582 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 2 hr, 24 min, 23 sec
3m
OCH3
172.879
159.063
152.506
144.117
136.557
133.136
129.571
129.342
127.667
126.478
124.861
124.474
113.679
111.288
77.415
76.986
76.571
72.261
55.024
52.347
46.191
14.007
13.634
ppm

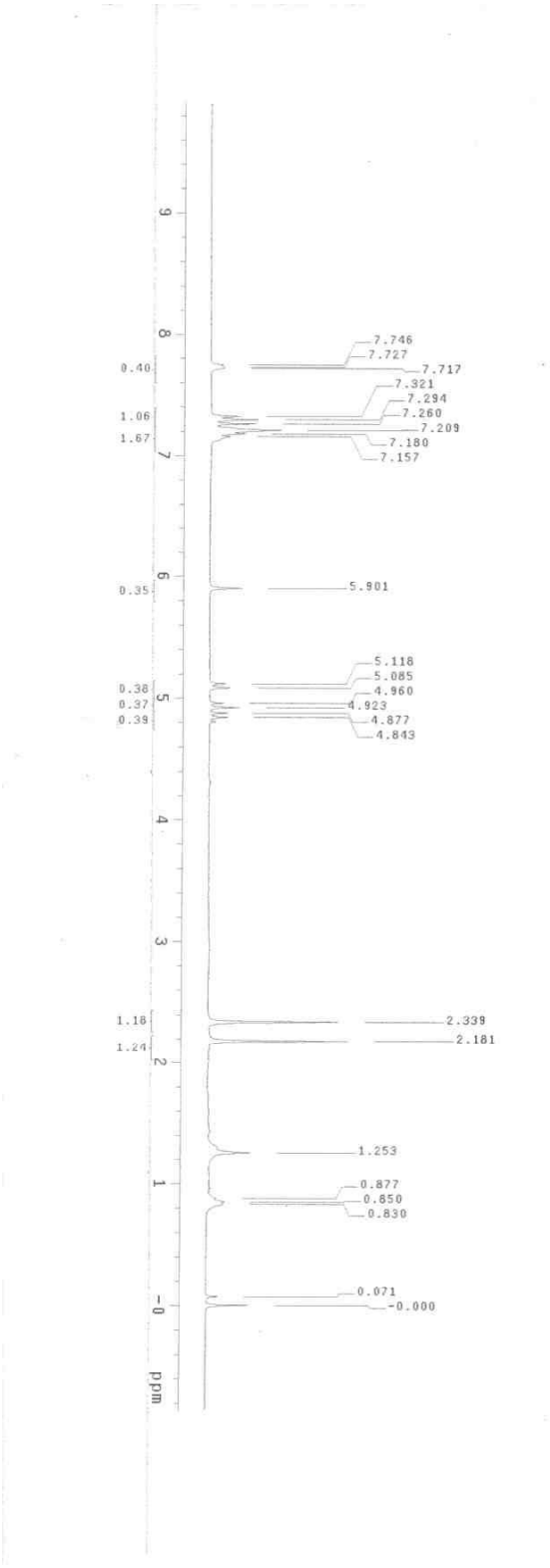

IJ-1-50C
3n
Cl
S
OH
N
N
O
7.744
7.748
7.370
7.342
7.311
7.258
7.210
7.200
7.156
7.132
7.110
7.092
5.908
5.541
5.505
5.185
4.999
4.964
4.930
3.357
3.334
2.343
2.181
1.681
-0.000
ppm

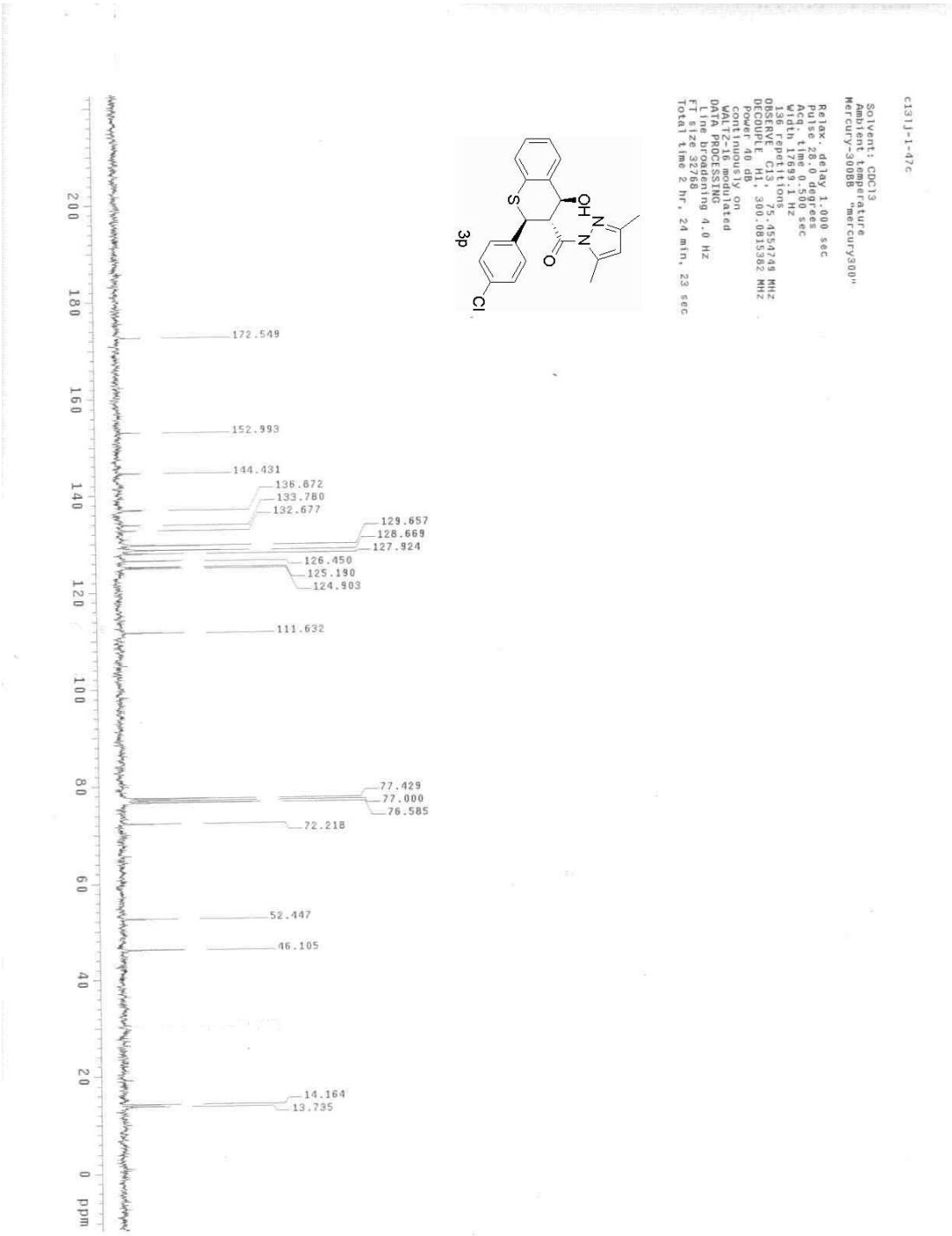

c131j-1-50c
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.500 sec
Width 17699.1 Hz
88 repetitions
OBSERVE C13, 75.4552999 MHz
DECOUPLE H1, 300.0815382 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 2 hr, 24 min, 23 sec
3n
Cl
S
OH
N
N
O
220
200
180
160
140
120
100
80
60
40
20
ppm
172.635
152.907
144.460
137.202
136.128
133.966
132.592
129.728
128.941
128.683
127.838
127.080
126.450
125.605
125.090
111.517
77.487
77.072
76.642
72.290
51.831
42.511
14.150
13.735

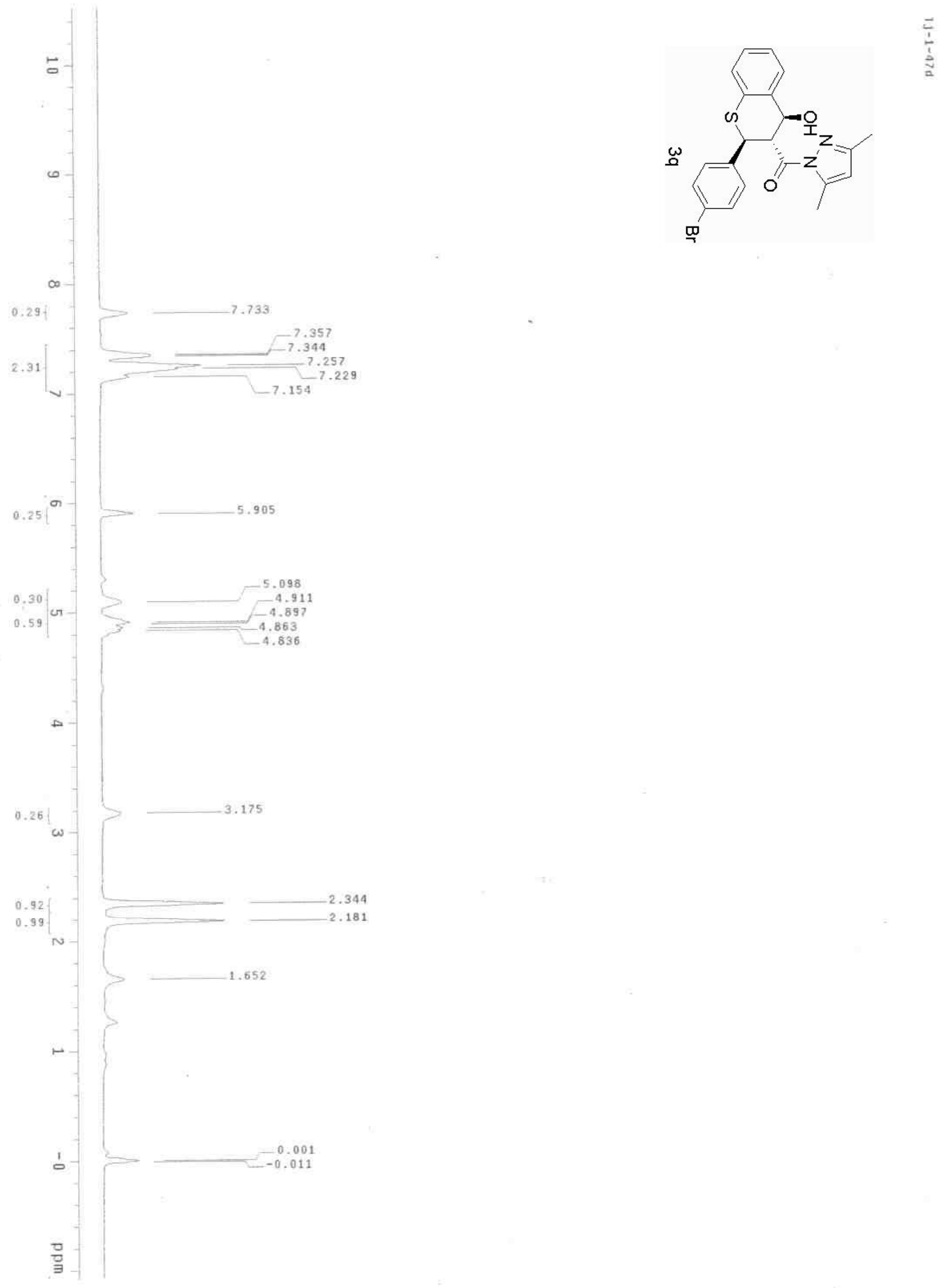

7.727
7.707
7.699
7.399
7.253
7.227
7.210
7.199
7.187
7.179
7.144
7.123
5.867
5.119
4.874
4.843
2.317
2.176
1.298
1.253
0.876
0.852
0.829
2.40
2.74
12.26
2.02
2.50
4.55
2.30
6.76
7.22
ppm

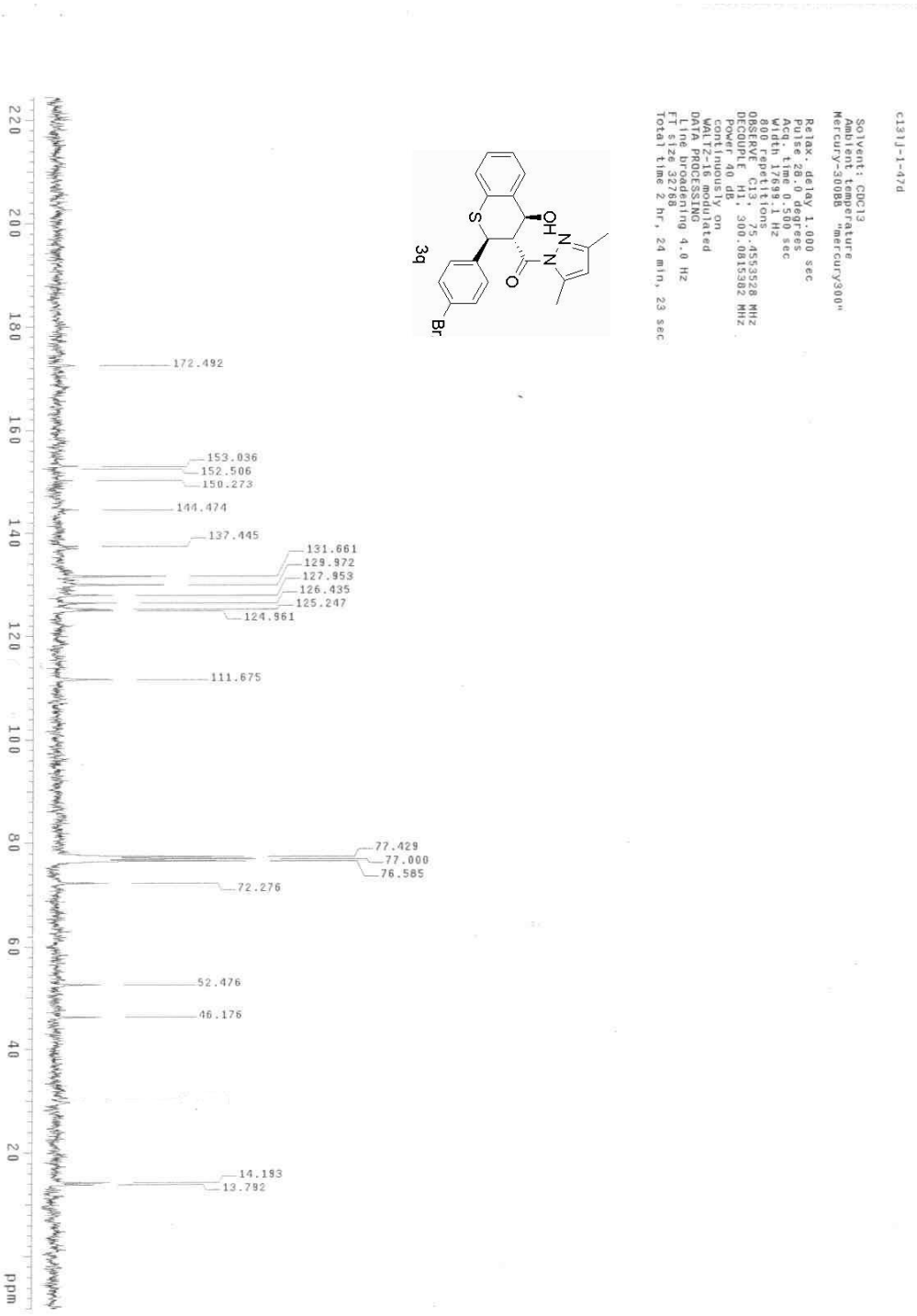

3o
S
OH
N
N
O
Cl

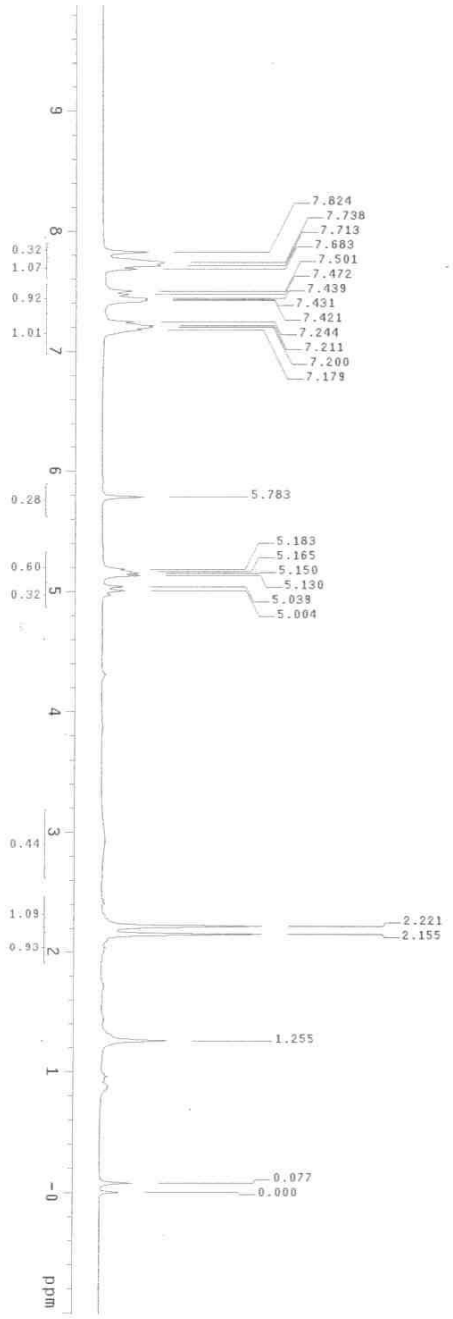

c131j-1-55b
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.500 sec
Width 17699.1 Hz
136 repetitions
OBSERVE C13, 75.4552899 MHz
DECOUPLE H1, 300.0815382 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 2 hr, 24 min, 23 sec
3o
S
OH
N
N
O
Cl
172.320
152.735
144.059
139.764
136.543
133.894
132.248
129.342
128.340
128.010
127.652
126.192
124.961
124.674
111.331
77.172
76.757
76.327
71.760
52.118
46.219
13.835
13.448
220
200
180
160
140
120
100
80
60
40
20
ppm

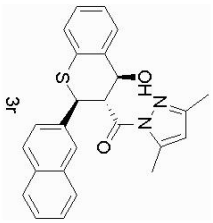

dxq-1-47c
3p
7.746
7.727
7.717
7.321
7.294
7.260
7.209
7.180
7.157
5.901
5.118
5.085
4.960
4.923
4.877
4.843
2.339
2.181
1.253
0.877
0.850
0.830
0.071
-0.000
0.40
1.06
1.67
0.35
0.38
0.37
0.39
1.18
1.24
ppm


c13j-1-47c
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degree
Acq. time 1.090 sec
Width 17699.1 Hz
136 repetitions
OBSERVE C13, 75.4554749 MHz
DECOUPLE H1, 300.0815382 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 2 hr, 24 min, 23 sec
3p
OH
N
N
O
S
Cl
172.549
152.993
144.431
136.672
133.780
132.677
129.657
128.669
127.924
126.450
125.190
124.903
111.632
77.429
77.000
76.585
72.218
52.447
46.105
14.164
13.735
200
180
160
140
120
100
80
60
40
20
0
ppm

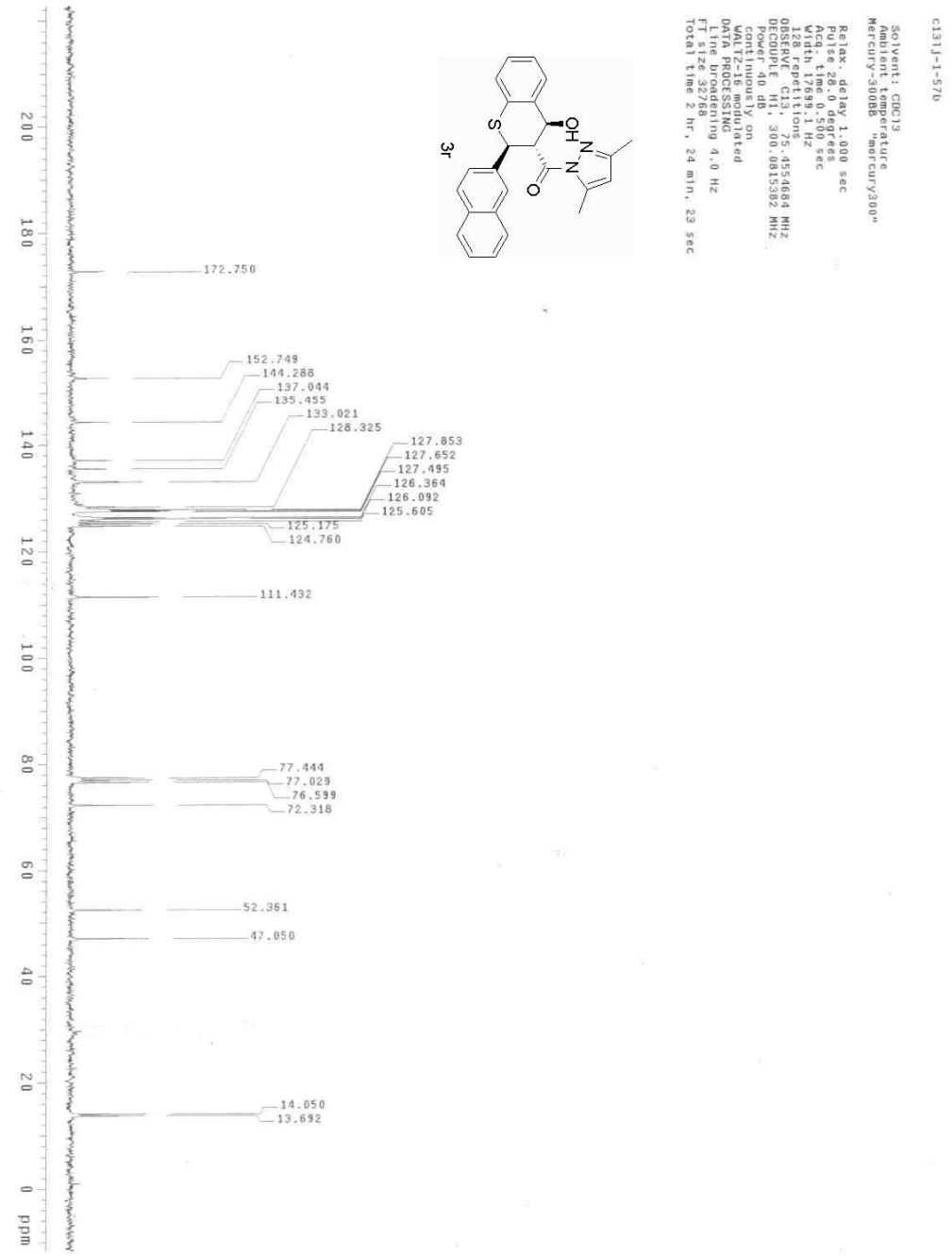


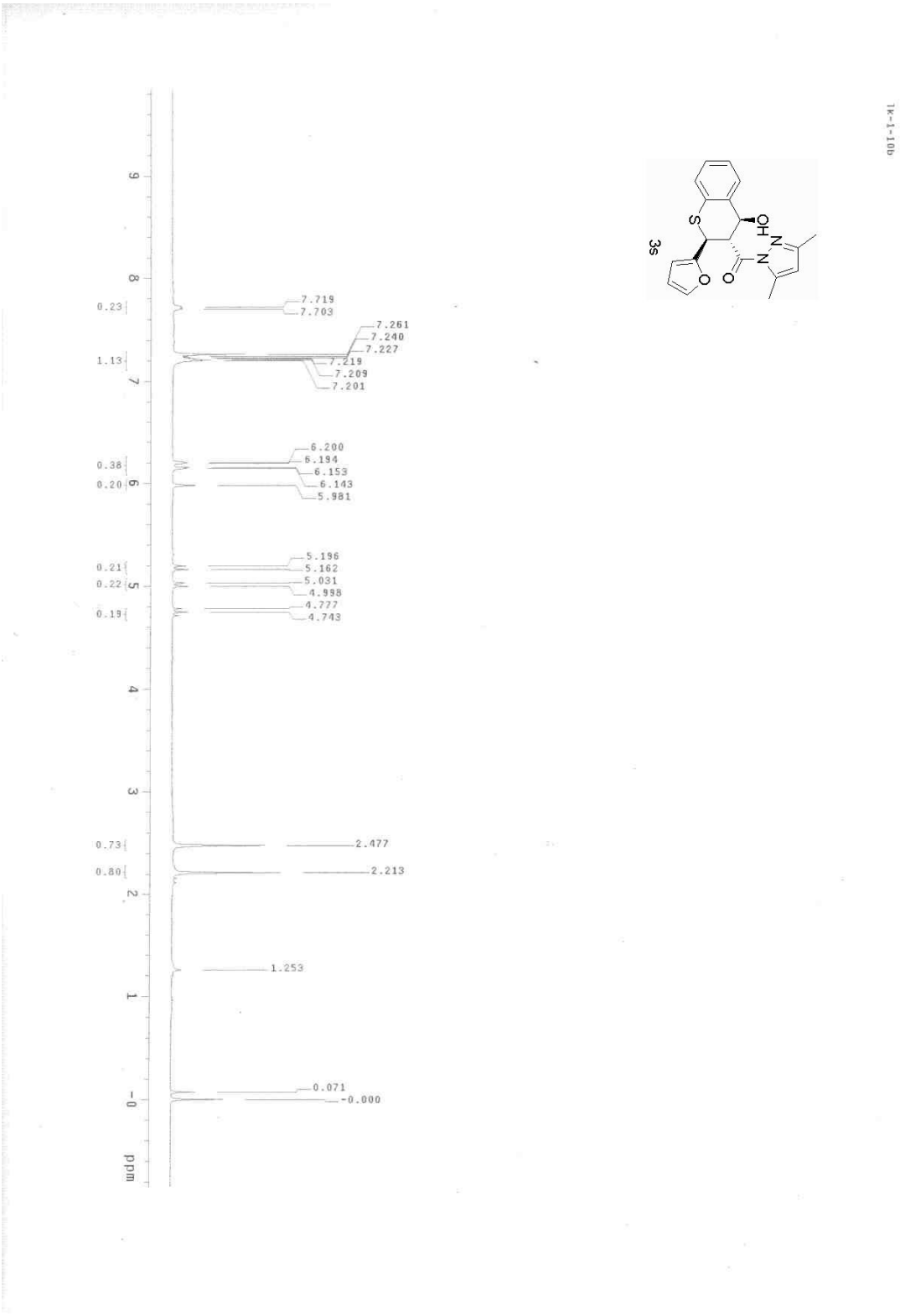

c13lj-1-47d
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.500 sec
Width 17699.1 Hz
800 repetitions
OBSERVE C13, 75.455528 MHz
DECOUPLE H1, 300.081502 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 2 hr, 24 min, 23 sec
3q
S
OH
N
N
O
Br
172.492
153.036
152.506
150.273
144.474
137.445
131.661
129.972
127.953
126.435
125.247
124.961
111.675
77.429
77.000
76.585
72.276
52.476
46.176
14.193
13.792
ppm

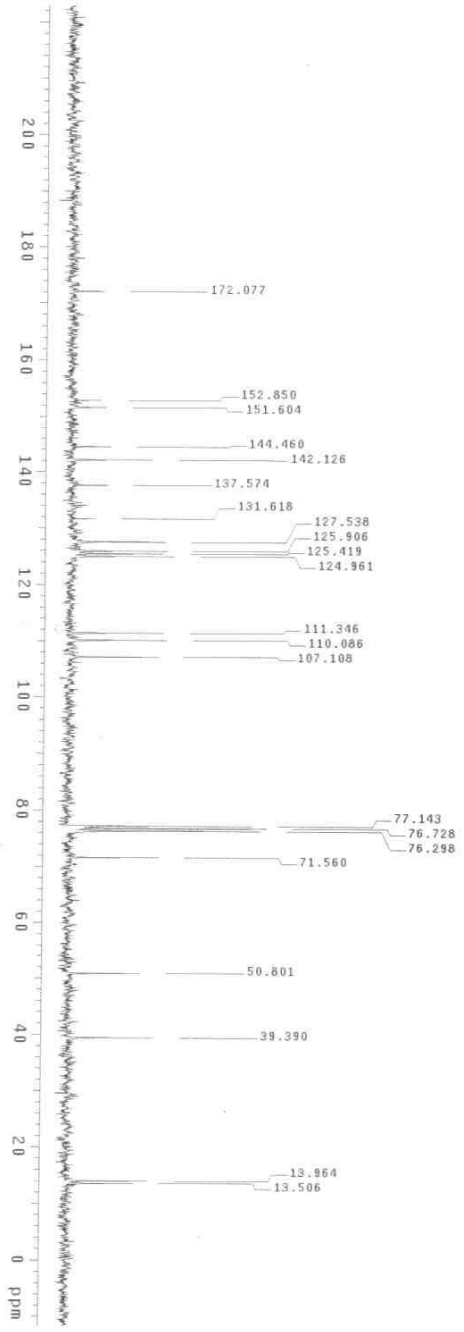

7.824
7.738
7.713
7.683
7.501
7.472
7.439
7.431
7.421
7.244
7.211
7.200
7.179
5.783
5.183
5.165
5.150
5.130
5.039
5.004
2.221
2.155
1.255
0.077
0.000
0.32
1.07
0.92
1.01
0.28
0.60
0.32
0.44
1.09
0.93
ppm

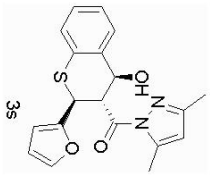

3r
OH
S
N
N
O

1j-1-57b


c131j-1-57b
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.500 sec
Width 17699.1 Hz
128 repetitions
OBSERVE C13, 75.4554684 MHz
DECOUPLE H1, 300.0015302 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 2 hr, 24 min, 23 sec
3r
OH
N
N
S
O
172.750
152.749
144.288
137.044
135.455
133.021
128.325
127.853
127.652
127.495
126.364
126.092
125.605
125.175
124.760
111.432
77.444
77.029
76.599
72.318
52.361
47.050
14.050
13.692
200
180
160
140
120
100
80
60
40
20
0
ppm


1k-1-10b
3s
OH
N
N
S
O
O
7.719
7.703
7.261
7.240
7.227
7.219
7.209
7.201
6.200
6.194
6.153
6.143
5.981
5.196
5.162
5.031
4.998
4.777
4.743
2.477
2.213
1.253
0.071
-0.000
0.23
1.13
0.38
0.20
0.21
0.22
0.19
0.73
0.80
ppm

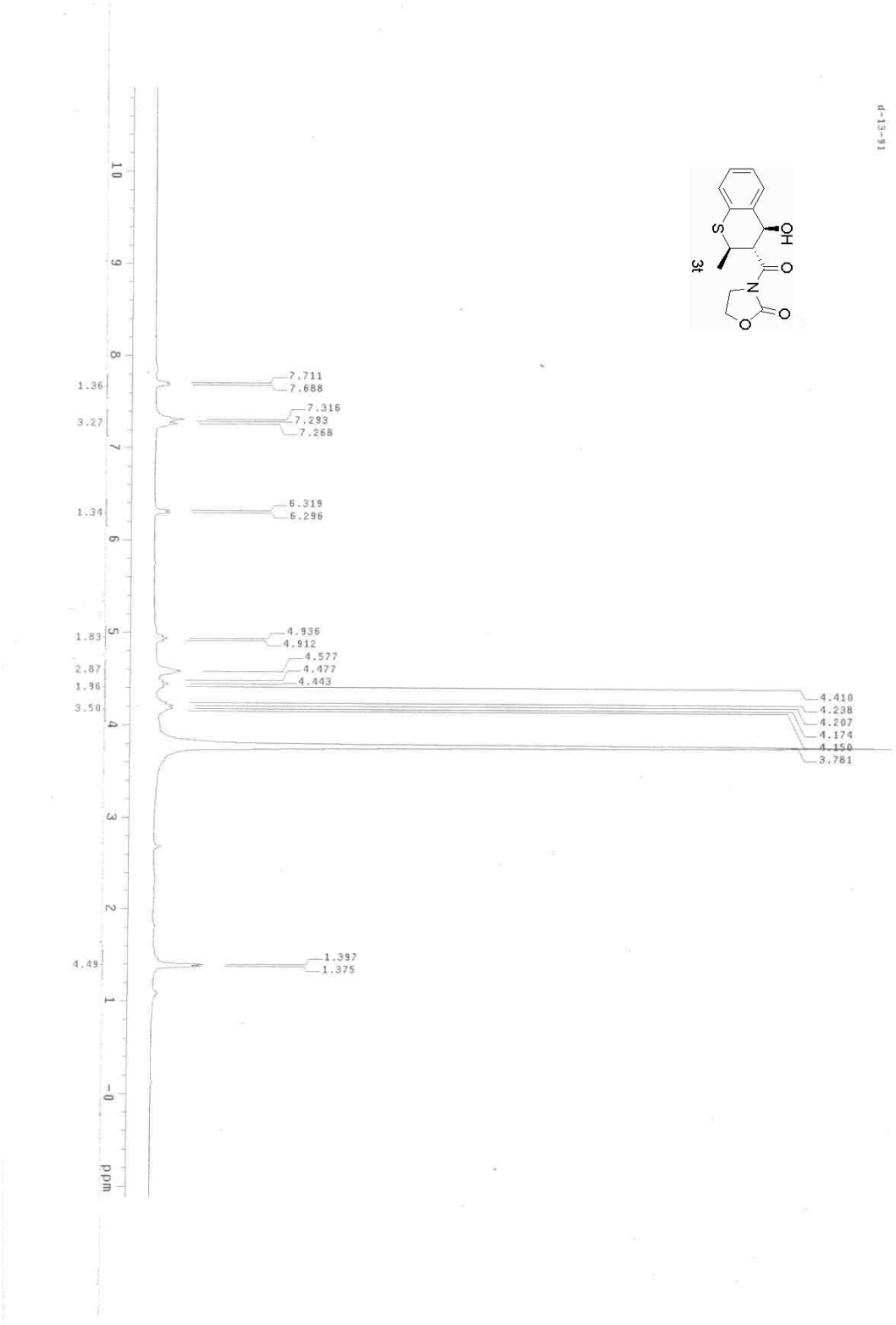


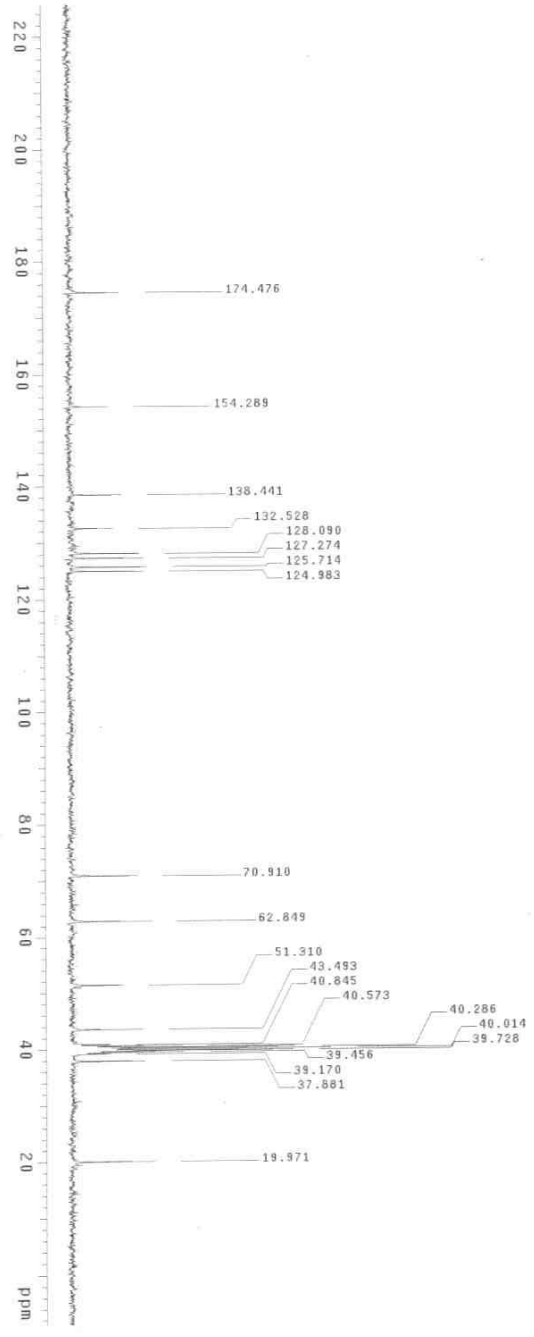


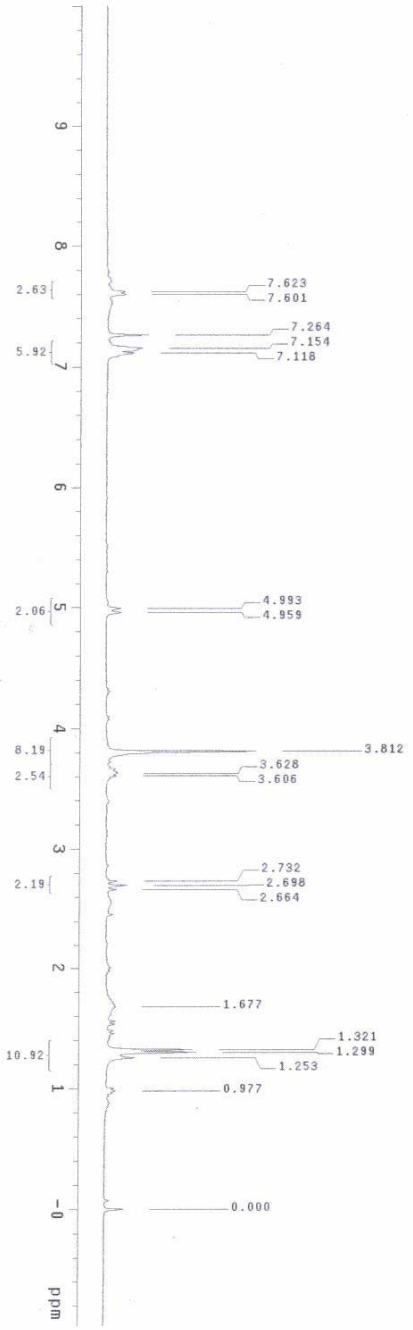
c131r-1-10b

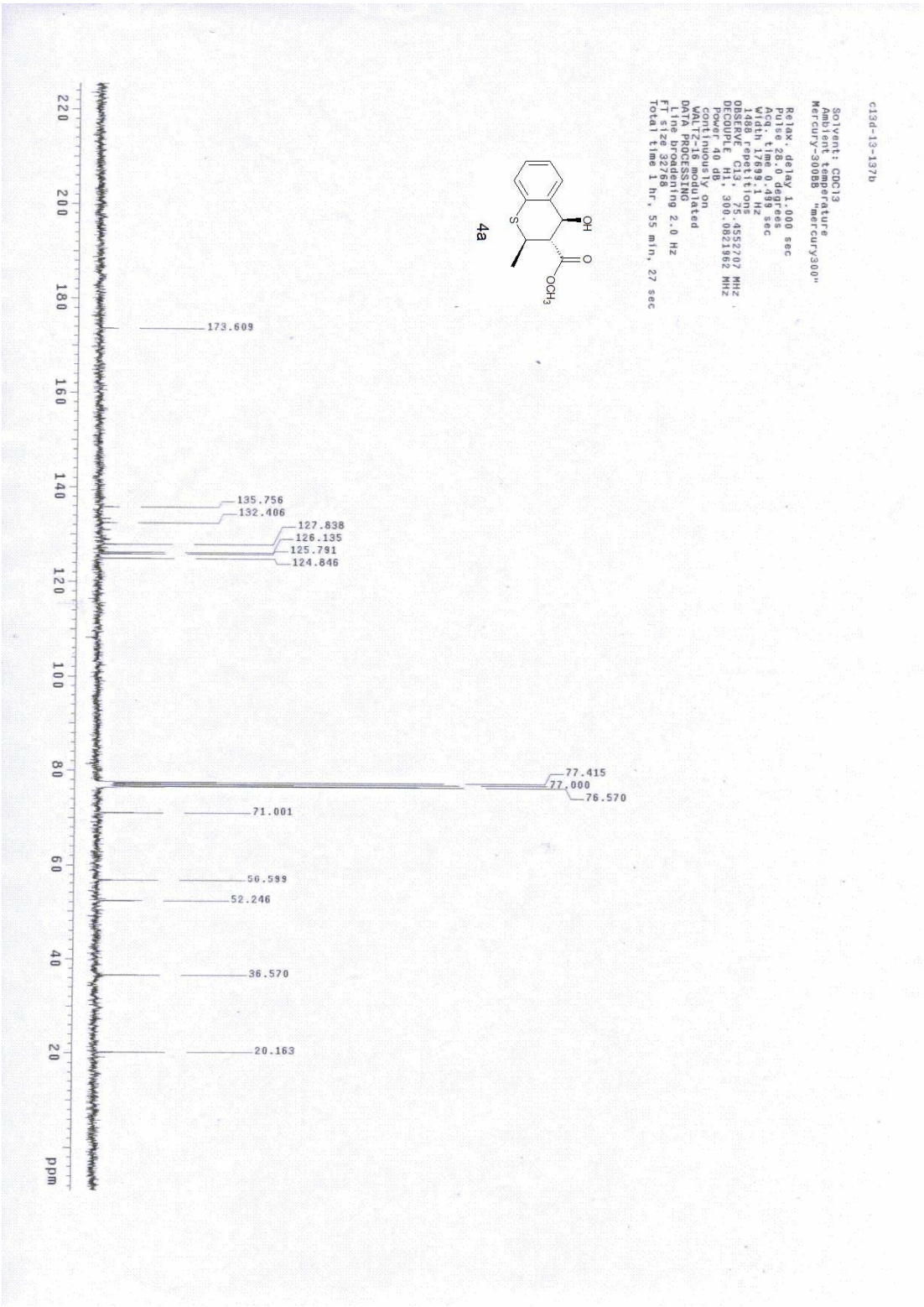
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"

Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.500 sec
Width 17699.1 Hz
160 repetitions
OBSERVE C13, 75.4554705 MHz
DECOUPLE H1, 300.0815382 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 2 hr, 24 min, 23 sec


d-13-91
3t
S
OH
O
N
O
O
1.36
3.27
1.34
1.83
2.87
1.96
3.50
4.49
7.711
7.688
7.316
7.293
7.268
6.319
6.296
4.936
4.912
4.577
4.477
4.443
4.410
4.238
4.207
4.174
4.150
3.781
1.397
1.375
ppm


dxq-C-120104
Solvent: DMSO
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.502 sec
Width 75189.1 Hz
128 repetitions
OBSERVE C13, 75.4556134 MHz
DECOUPLE H1, 300.0839276 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 19 min, 27 sec
3t
OH
O
N
O
O
S
174.476
154.289
138.441
132.528
128.090
127.274
125.714
124.983
70.910
62.849
51.310
43.493
40.845
40.573
40.286
40.014
39.728
39.456
39.170
37.881
19.971
220
200
180
160
140
120
100
80
60
40
20
ppm


d-13-137b
4a
OH
O
S
OCH3
2.63
5.92
2.06
8.19
2.54
2.19
10.92
7.623
7.601
7.264
7.154
7.118
4.993
4.959
3.812
3.628
3.606
2.732
2.698
2.664
1.677
1.321
1.299
1.253
0.977
0.000
ppm


c13d-13-137b
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.499 sec
Width 17699.1 Hz
1488 repetitions
OBSERVE C13, 75.4552707 MHz
DECOUPLE H1, 300.0821962 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 32768
Total time 1 hr, 55 min, 27 sec
4a
OH
O
OCH3
S
173.609
135.756
132.406
127.838
126.135
125.791
124.846
77.415
77.000
76.570
71.001
56.599
52.246
36.570
20.163
ppm

VIII. HPLC Chromatograms

Data File D:\LC\FX\DATA\FX-4-18B\FX-4-18B-9 2011-09-27 16-33-09\012-0201.D
Sample Name: FX-4-18b

```
==================================================================
Acq. Operator   : FX                          Seq. Line :   2
Acq. Instrument : Instrument 1                 Location : Vial 12
Injection Date  : 9/27/2011 4:45:40 PM             Inj :   1
                                              Inj Volume : 5 µl
Acq. Method     : D:\LC\FX\Data\FX-4-18B\FX-4-18B-9 2011-09-27 16-33-09\ASH-10-90-05ML-
                  254NM.M
Last changed    : 9/27/2011 5:21:15 PM by FX
                  (modified after loading)
Analysis Method : D:\LC\FX\DATA\FX-4-18B\FX-4-18B-9 2011-09-27 16-33-09\012-0201.D\DA.M (
                  ASH-10-90-05ML-254NM.M)
Last changed    : 10/5/2011 10:36:42 PM by dxq
                  (modified after loading)
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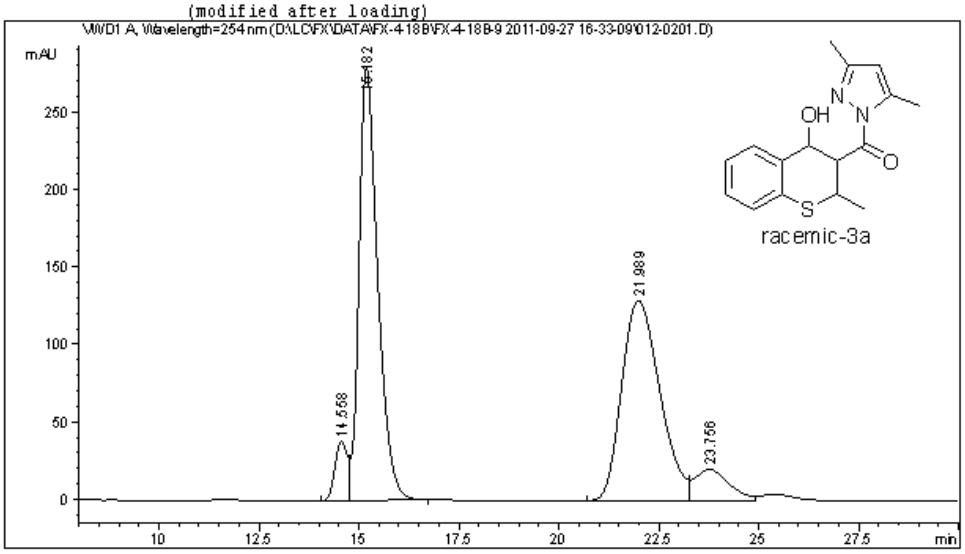



```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs

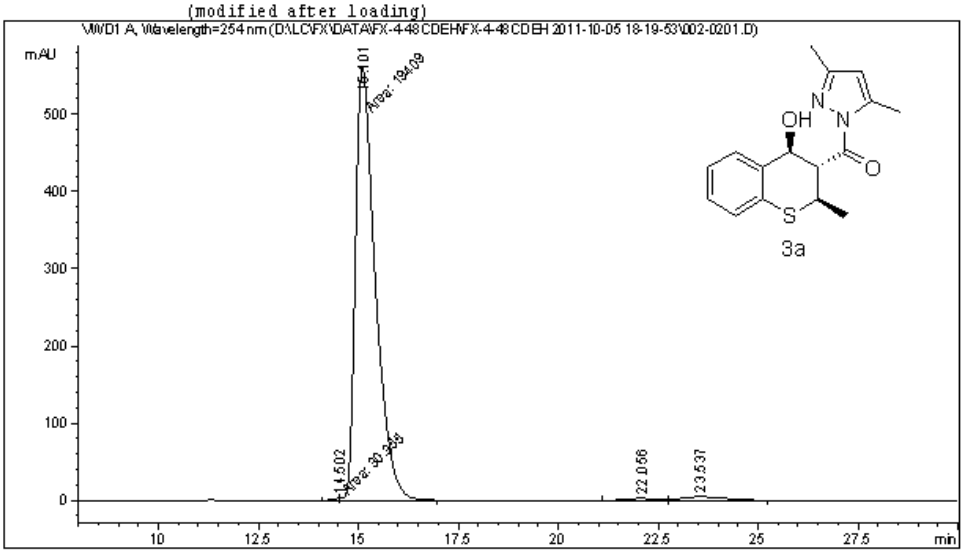

Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s    [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  14.558 BV    0.3377  826.58118   37.85433   4.2004
   2  15.182 VB    0.4909 9070.29004  279.33612  46.0916
   3  21.989 BV    1.0405 8588.54590  129.20874  43.6435
   4  23.756 VV    0.8653 1193.43799   20.24796   6.0646

Totals :                  1.96789e4   466.64716
```

Data File D:\LC\FX\DATA\FX-4-48CDEH\FX-4-48CDEH 2011-10-05 18-19-53\002-0201.D
Sample Name: FX-4-48Cb

Acq. Operator	: FX	Seq. Line	: 2
Acq. Instrument	: Instrument 1	Location	: Vial 2
Injection Date	: 10/5/2011 6:32:27 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\FX\Data\FX-4-48CDEH\FX-4-48CDEH 2011-10-05 18-19-53\ASH-10-90-05ML-254NM-30MIN.M		
Last changed	: 9/28/2011 11:47:06 AM by thl		
Analysis Method	: D:\LC\FX\DATA\FX-4-48CDEH\FX-4-48CDEH 2011-10-05 18-19-53\002-0201.D\DA.M (ASH-10-90-05ML-254NM-30MIN.M)		
Last changed	: 10/5/2011 10:33:39 PM by dxq (modified after loading)		



Area Percent Report

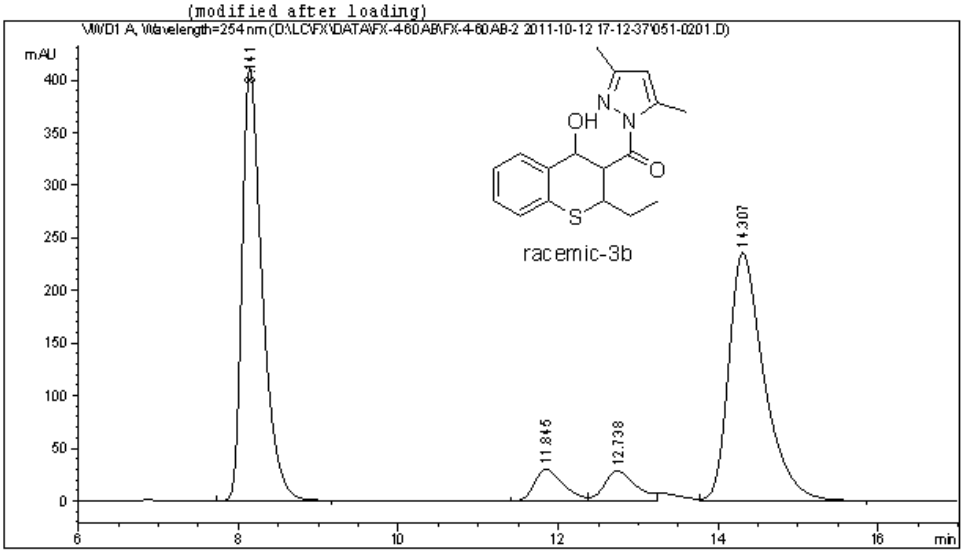
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	14.502	MF	0.1865	30.35498	2.71309	0.1522
2	15.101	FM	0.5769	1.94090e4	560.69159	97.2966
3	22.056	BV	0.7669	157.12704	2.63550	0.7877
4	23.537	VB	0.9386	351.79373	4.76448	1.7635
Totals :				1.99483e4	570.80466	

Data File D:\LC\FX\DATA\FX-4-60AB\FX-4-60AB-2 2011-10-12 17-12-37\051-0201.D
Sample Name: FX-4-60Ab

Acq. Operator	: FX	Seq. Line	: 2
Acq. Instrument	: Instrument 1	Location	: Vial 51
Injection Date	: 10/12/2011 5:25:29 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\FX\Data\FX-4-60AB\FX-4-60AB-2 2011-10-12 17-12-37\ADH-10-90-1ML-254NM.M		
Last changed	: 10/12/2011 5:44:42 PM by FX (modified after loading)		
Analysis Method	: D:\LC\FX\DATA\FX-4-60AB\FX-4-60AB-2 2011-10-12 17-12-37\051-0201.D\DA.M (ADH-10-90-1ML-254NM.M)		
Last changed	: 10/13/2011 12:04:17 PM by FX (modified after loading)		



Area Percent Report

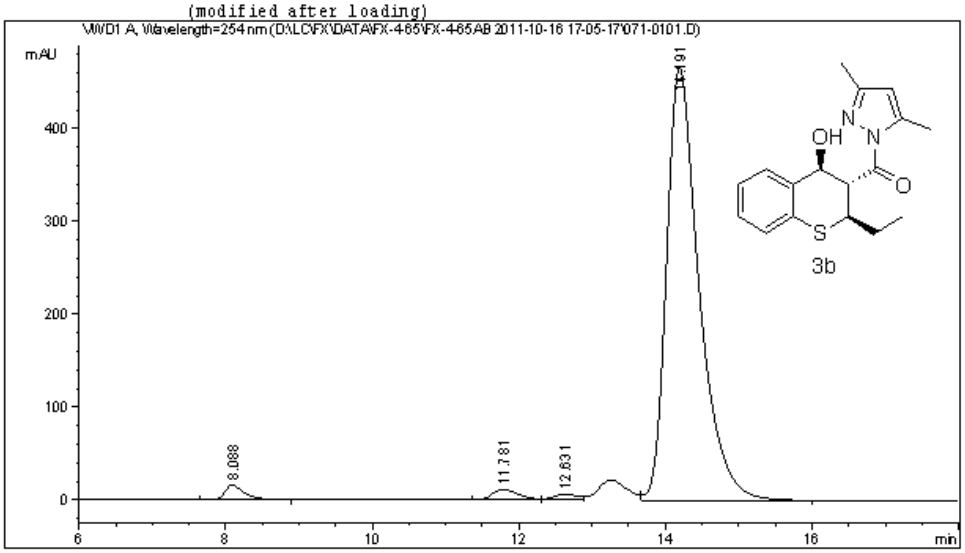
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	8.141	BB	0.2605	7189.43945	411.68027	45.0744
2	11.845	BV	0.3717	753.37109	30.44006	4.7233
3	12.738	VV	0.4040	776.10846	28.60009	4.8658
4	14.307	VB	0.4583	7231.22803	235.79341	45.3364
Totals :				1.5950le4	706.51383	

Data File D:\LC\FX\DATA\FX-4-65\FX-4-65AB 2011-10-16 17-05-17\071-0101.D
Sample Name: fx-4-65ab

Acq. Operator	: dxq	Seq. Line	: 1
Acq. Instrument	: Instrument 1	Location	: Vial 71
Injection Date	: 10/16/2011 5:06:30 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\FX\Data\FX-4-65\FX-4-65AB 2011-10-16 17-05-17\ADH-10-90-1ML-254NM.M		
Last changed	: 10/16/2011 5:06:40 PM by dxq (modified after loading)		
Analysis Method	: D:\LC\FX\DATA\FX-4-65\FX-4-65AB 2011-10-16 17-05-17\071-0101.D\DA.M (ADH-10-90-1ML-254NM.M)		
Last changed	: 10/18/2011 3:18:36 PM by FX (modified after loading)		



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU*s	Height [mAU]	Area %
1	8.088	BB	0.2755	308.92929	16.36662	2.0444
2	11.781	BV	0.3713	289.13525	11.75995	1.9134
3	12.631	VV	0.3463	151.49113	6.67530	1.0025
4	14.191	VB	0.4594	1.43614e4	466.79181	95.0396
Totals :				1.51109e4	501.59369	

Data File D:\LC\FX\DATA\FX-4-63-64AB\FX-4-63-64AB-1 2011-10-14 14-23-40\053-0301.D
Sample Name: FX-4-63AB

```
==================================================================
Acq. Operator    : FX                          Seq. Line :   3
Acq. Instrument  : Instrument 1                 Location : Vial 53
Injection Date   : 10/14/2011 2:58:47 PM              Inj :   1
                                               Inj Volume : 5 µl
Acq. Method      : D:\LC\FX\Data\FX-4-63-64AB\FX-4-63-64AB-1 2011-10-14 14-23-40\0JH-10-90-
                   05ML-254NM.M
Last changed     : 10/14/2011 3:33:23 PM by FX
                   (modified after loading)
Analysis Method  : D:\LC\FX\DATA\FX-4-63-64AB\FX-4-63-64AB-1 2011-10-14 14-23-40\053-0301.D\
                   DA.M (0JH-10-90-05ML-254NM.M)
Last changed     : 10/18/2011 3:12:38 PM by FX
                   (modified after loading)
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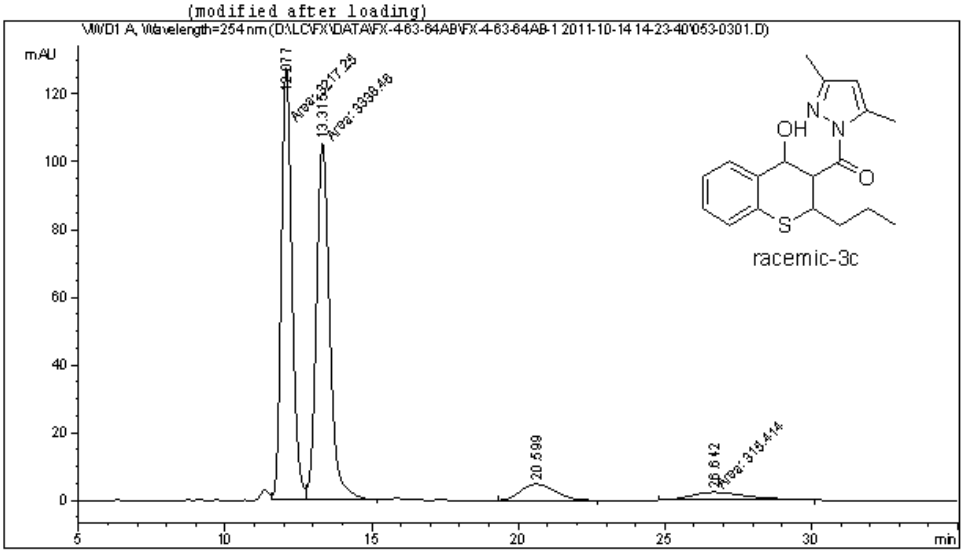
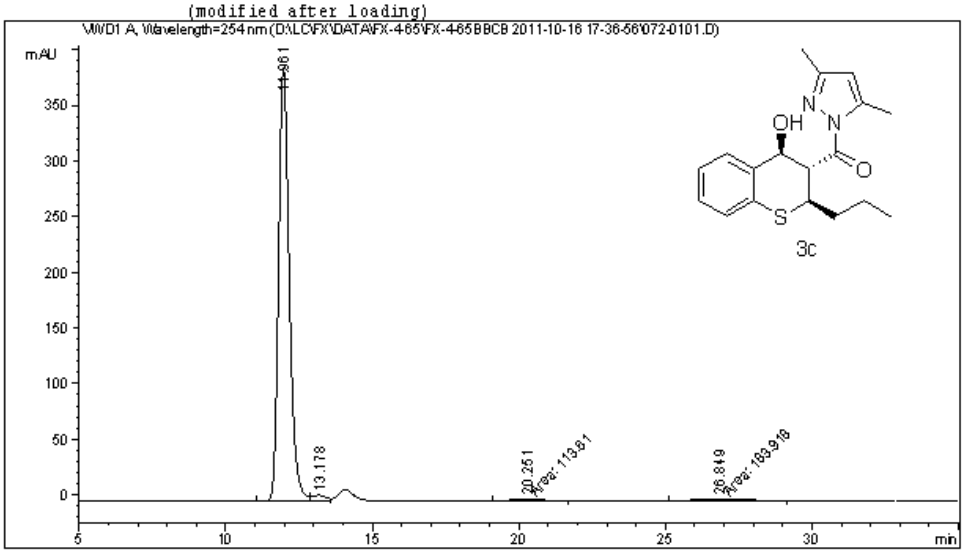



```
==================================================================
                      Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  12.077 FM    0.4198 3217.25391  127.73701  44.2871
   2  13.315 FM    0.5287 3338.45825  105.24897  45.9555
   3  20.599 BB    0.9790  393.42123    4.73783   5.4156
   4  26.642 MM    2.2470  315.41403    2.33952   4.3418

Totals :                  7264.54742  240.06333
```

Data File D:\LC\FX\DATA\FX-4-65\FX-4-65BBCB 2011-10-16 17-36-56\072-0101.D
Sample Name: fx-4-65bb

Acq. Operator	: dxq	Seq. Line	: 1
Acq. Instrument	: Instrument 1	Location	: Vial 72
Injection Date	: 10/16/2011 5:38:15 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\FX\Data\FX-4-65\FX-4-65BBCB 2011-10-16 17-36-56\0JH-10-90-05ML-254NM-30MIN.M		
Last changed	: 10/16/2011 6:07:48 PM by dxq (modified after loading)		
Analysis Method	: D:\LC\FX\DATA\FX-4-65\FX-4-65BBCB 2011-10-16 17-36-56\072-0101.D\DA.M (0JH-10-90-05ML-254NM-30MIN.M)		
Last changed	: 10/18/2011 3:22:15 PM by FX (modified after loading)		



Area Percent Report

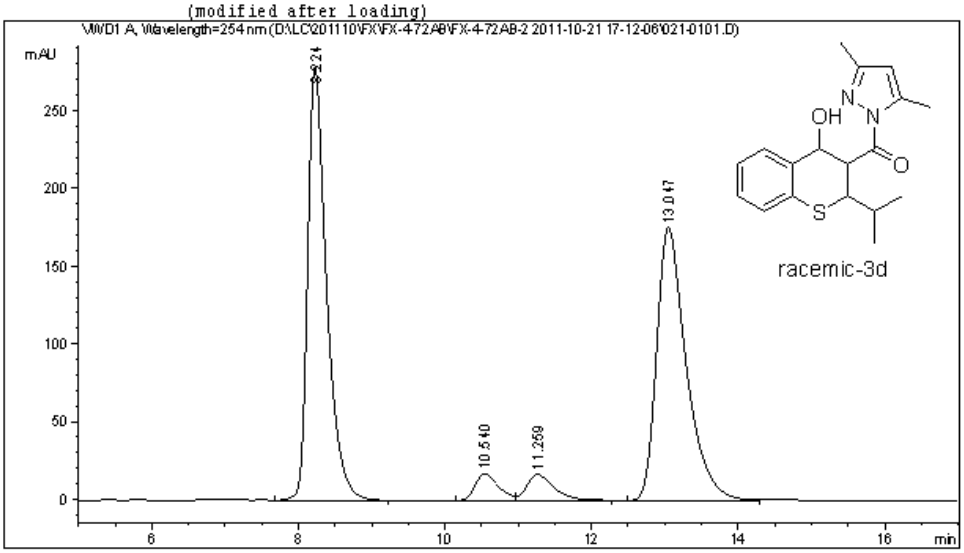
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	11.961	BV	0.3876	9824.30566	389.35544	95.5955
2	13.178	VV	0.4606	155.11661	4.90540	1.5094
3	20.251	MM	1.1559	113.60997	1.63817	1.1055
4	26.849	MM	1.9839	183.91774	1.54506	1.7896
Totals :				1.02769e4	397.44407	

Data File D:\LC\201110\FX\FX-4-72AB\FX-4-72AB-2 2011-10-21 17-12-06\021-0101.D
Sample Name: FX-4-72Ab

Acq. Operator	: FX	Seq. Line	: 1
Acq. Instrument	: Instrument 1	Location	: Vial 21
Injection Date	: 10/21/2011 5:13:38 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201110\FX\FX-4-72AB\FX-4-72AB-2 2011-10-21 17-12-06\ADH-10-90-1ML-254NM.M		
Last changed	: 9/27/2011 1:52:39 PM by THL		
Analysis Method	: D:\LC\201110\FX\FX-4-72AB\FX-4-72AB-2 2011-10-21 17-12-06\021-0101.D\DA.M (ADH-10-90-1ML-254NM.M)		
Last changed	: 10/21/2011 5:43:09 PM by HZL (modified after loading)		



Area Percent Report

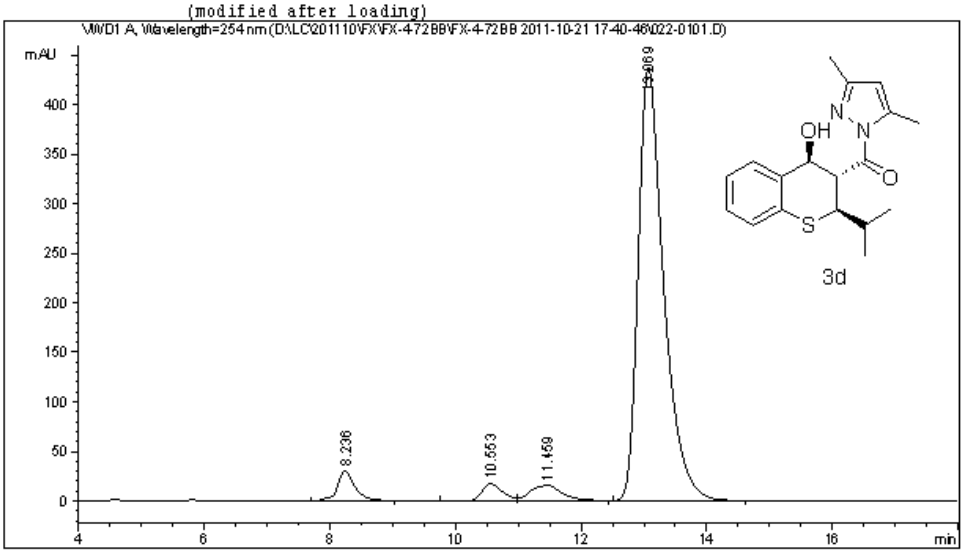
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	8.224	VB	0.2653	4981.61133	278.70477	46.4797
2	10.540	BV	0.3283	380.28021	17.46513	3.5481
3	11.259	VB	0.3842	436.08261	16.72797	4.0688
4	13.047	BB	0.4187	4919.84131	175.52832	45.9034
Totals :				1.07178e4	488.42619	

Data File D:\LC\201110\FX\FX-4-72BB\FX-4-72BB 2011-10-21 17-40-46\022-0101.D
Sample Name: FX-4-72Bb

Acq. Operator	: FX	Seq. Line	: 1
Acq. Instrument	: Instrument 1	Location	: Vial 22
Injection Date	: 10/21/2011 5:42:36 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201110\FX\FX-4-72BB\FX-4-72BB 2011-10-21 17-40-46\ADH-10-90-1ML-254NM.M		
Last changed	: 10/21/2011 5:45:16 PM by FX (modified after loading)		
Analysis Method	: D:\LC\201110\FX\FX-4-72BB\FX-4-72BB 2011-10-21 17-40-46\022-0101.D\DA.M (ADH-10-90-1ML-254NM.M)		
Last changed	: 10/22/2011 1:27:55 PM by FX (modified after loading)		



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	8.236	BB	0.2804	585.21027	30.53090	4.2252
2	10.553	BV	0.3375	398.13519	17.64404	2.8745
3	11.459	VB	0.5540	552.38464	15.61468	3.9882
4	13.069	BB	0.4204	1.23147e4	437.13513	88.9121
Totals :				1.38504e4	500.92475	

Data File D:\LC\FX\DATA\FX-4-63-64AB\FX-4-63-64AB-1 2011-10-14 14-23-40\054-0401.D
Sample Name: FX-4-64AB

```
==================================================================
Acq. Operator   : FX                          Seq. Line :   4
Acq. Instrument : Instrument 1                 Location : Vial 54
Injection Date  : 10/14/2011 3:35:29 PM              Inj :   1
                                             Inj Volume : 5 µl
Acq. Method     : D:\LC\FX\Data\FX-4-63-64AB\FX-4-63-64AB-1 2011-10-14 14-23-40\0JH-10-90-
                  05ML-254NM.M
Last changed    : 10/14/2011 4:02:38 PM by FX
                  (modified after loading)
Analysis Method : D:\LC\FX\DATA\FX-4-63-64AB\FX-4-63-64AB-1 2011-10-14 14-23-40\054-0401.D\
                  DA.M (0JH-10-90-05ML-254NM.M)
Last changed    : 10/18/2011 3:15:33 PM by FX
                  (modified after loading)
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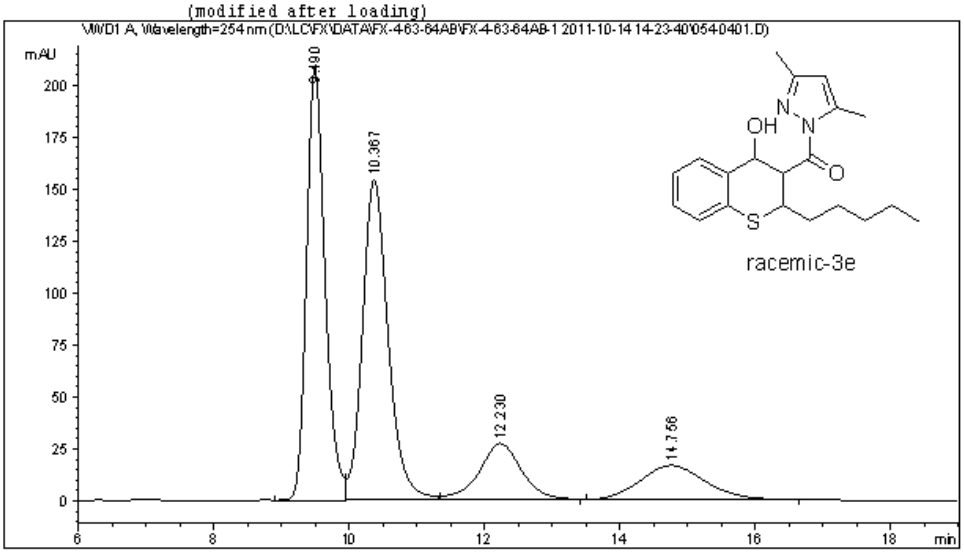


```
==================================================================
                      Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1   9.490 VV    0.2905 3965.47144  208.28885  38.4115
   2  10.367 VB    0.4020 4066.07324  154.34161  39.3859
   3  12.230 BB    0.6497 1183.71899   27.07998  11.4661
   4  14.756 BB    0.9984 1108.40100   16.45676  10.7365

Totals :                  1.03237e4   406.16720
```

Data File D:\LC\FX\DATA\FX-4-65\FX-4-65BBCB 2011-10-16 17-36-56\073-0201.D
Sample Name: fx-4-65cb

```
=====================================================================
Acq. Operator   : dxq                          Seq. Line :   2
Acq. Instrument : Instrument 1                  Location : Vial 73
Injection Date  : 10/16/2011 6:14:42 PM              Inj :   1
                                               Inj Volume : 5 µl
Acq. Method     : D:\LC\FX\Data\FX-4-65\FX-4-65BBCB 2011-10-16 17-36-56\0JH-10-90-05ML-
                  254NM-20MIN.M
Last changed    : 10/16/2011 5:33:37 PM by dxq
Analysis Method : D:\LC\FX\DATA\FX-4-65\FX-4-65BBCB 2011-10-16 17-36-56\073-0201.D\DA.M (
                  0JH-10-90-05ML-254NM-20MIN.M)
Last changed    : 10/18/2011 3:27:34 PM by FX
                  (modified after loading)
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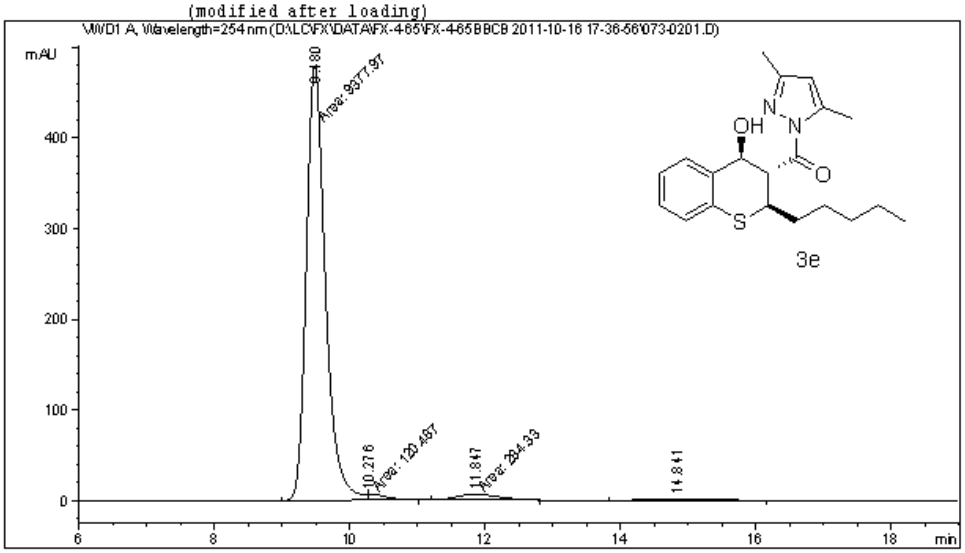
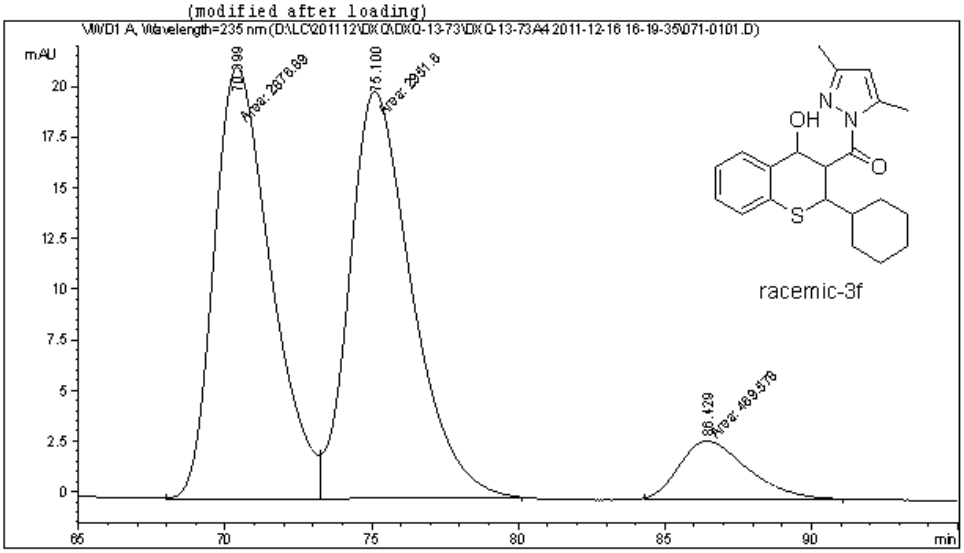



```
=====================================================================
                        Area Percent Report
=====================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1   9.480 MF    0.3271 9377.97168  477.77005  94.5068
   2  10.276 FM    0.3087  120.48733    6.50533   1.2142
   3  11.847 MM    0.7489  284.33029    6.32756   2.8653
   4  14.841 BV    0.7969  140.27965    2.15204   1.4137

Totals :                  9923.06895  492.75498
```

Data File D:\LC\201112\DXQ\DXQ-13-73\DXQ-13-73A4 2011-12-16 16-19-35\071-0101.D
Sample Name: dxq-13-73a

Acq. Operator	: LQH	Seq. Line	: 1
Acq. Instrument	: Instrument 1	Location	: Vial 71
Injection Date	: 12/16/2011 4:20:21 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201112\dxq\DXQ-13-73\DXQ-13-73A4 2011-12-16 16-19-35\ADH-5-95-025ML-235NM.M		
Last changed	: 9/26/2011 4:59:40 PM by THL		
Analysis Method	: D:\LC\201112\DXQ\DXQ-13-73\DXQ-13-73A4 2011-12-16 16-19-35\071-0101.D\DA.M (ADH-5-95-025ML-235NM.M)		
Last changed	: 12/18/2011 7:18:49 PM by thl (modified after loading)		



Area Percent Report

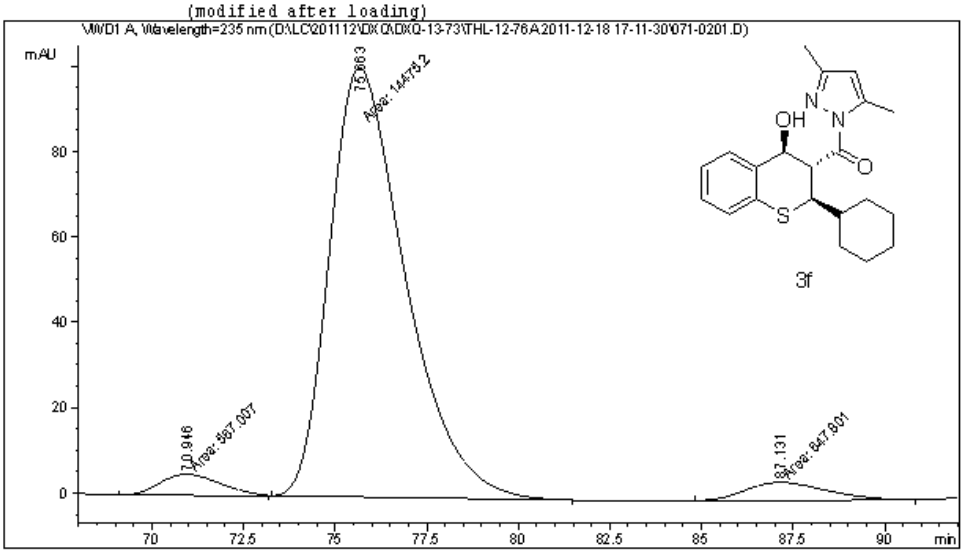
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=235 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	70.399	MF	2.2487	2876.88794	21.32232	45.6789
2	75.100	FM	2.4484	2951.60059	20.09208	46.8652
3	86.429	MM	2.7218	469.57779	2.87539	7.4559
Totals :				6298.06631	44.28978	

Data File D:\LC\201112\DXQ\DXQ-13-73\THL-12-76A 2011-12-18 17-11-30\071-0201.D
Sample Name: dxq-13-73c

Acq. Operator	: thl	Seq. Line	: 2
Acq. Instrument	: Instrument 1	Location	: Vial 71
Injection Date	: 12/18/2011 5:16:23 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201112\dxq\DXQ-13-73\THL-12-76A 2011-12-18 17-11-30\ADH-5-95-025ML-235NM.M		
Last changed	: 12/18/2011 5:16:48 PM by thl (modified after loading)		
Analysis Method	: D:\LC\201112\DXQ\DXQ-13-73\THL-12-76A 2011-12-18 17-11-30\071-0201.D\DA.M (ADH-5-95-025ML-235NM.M)		
Last changed	: 12/18/2011 7:21:08 PM by thl (modified after loading)		



Area Percent Report

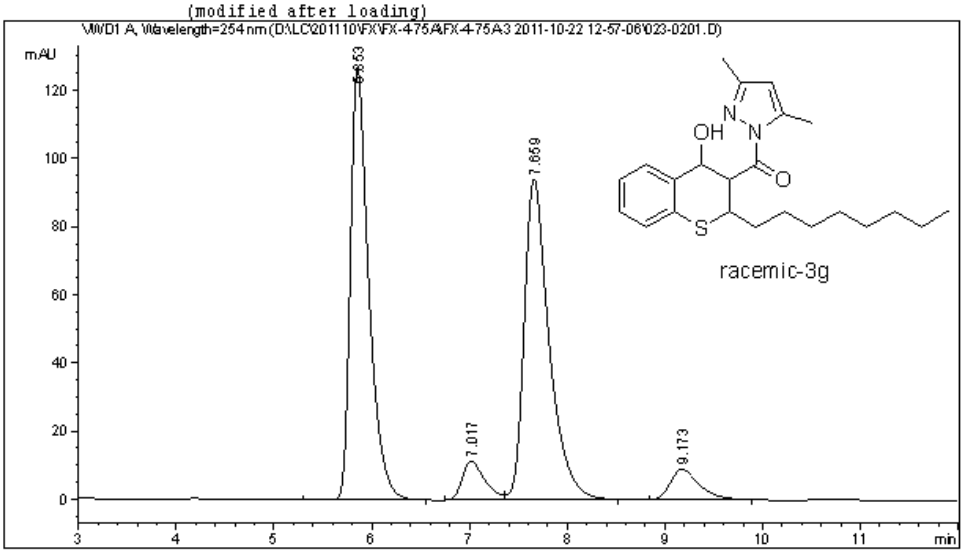
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=235 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	70.946	MM	1.9099	567.00677	4.94795	3.6138
2	75.663	MM	2.3996	1.44752e4	100.53846	92.2574
3	87.131	MM	2.5521	647.80060	4.23054	4.1288
Totals :				1.56900e4	109.71695	

Data File D:\LC\201110\FX\FX-4-75A\FX-4-75A-3 2011-10-22 12-57-06\023-0201.D
Sample Name: FX-4-75A

Acq. Operator	: FX	Seq. Line	: 2
Acq. Instrument	: Instrument 1	Location	: Vial 23
Injection Date	: 10/22/2011 1:09:32 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201110\FX\FX-4-75A\FX-4-75A-3 2011-10-22 12-57-06\ADH-10-90-1ML-254NM.M		
Last changed	: 9/27/2011 1:52:39 PM by THL		
Analysis Method	: D:\LC\201110\FX\FX-4-75A\FX-4-75A-3 2011-10-22 12-57-06\023-0201.D\DA.M (ADH-10-90-1ML-254NM.M)		
Last changed	: 10/22/2011 1:31:52 PM by FX (modified after loading)		



Area Percent Report

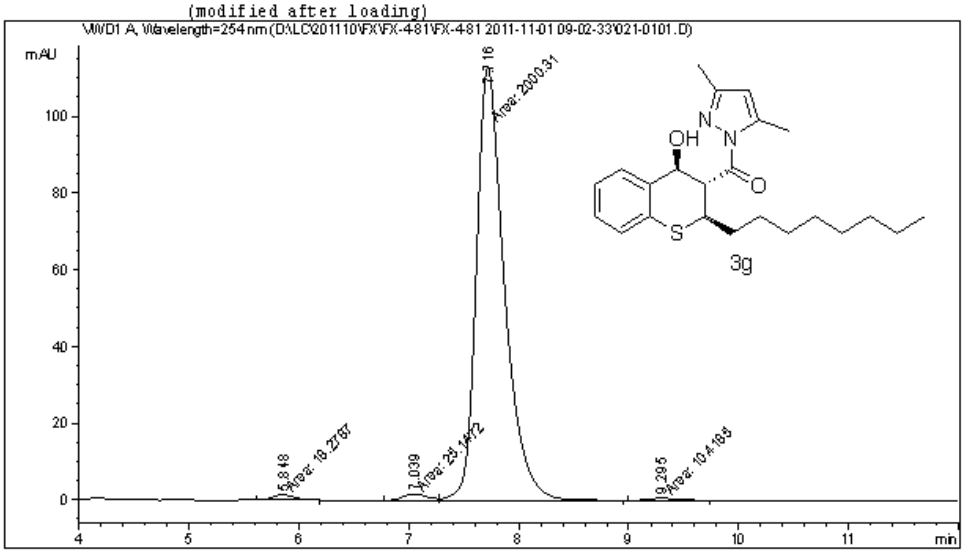
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	5.853	BB	0.1947	1659.81323	127.03289	44.8306
2	7.017	BV	0.2421	182.61031	11.40605	4.9322
3	7.659	VB	0.2643	1676.00305	94.21970	45.2679
4	9.173	BB	0.3068	183.98338	8.95162	4.9693
Totals :				3702.40997	241.61025	

Data File D:\LC\201110\FX\FX-4-81\FX-4-81 2011-11-01 09-02-33\021-0101.D
Sample Name: FX-4-81

Acq. Operator	: FX	Seq. Line	: 1
Acq. Instrument	: Instrument 1	Location	: Vial 21
Injection Date	: 11/1/2011 9:03:58 AM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201110\FX\FX-4-81\FX-4-81 2011-11-01 09-02-33\ADH-10-90-1ML-254NM.M		
Last changed	: 11/1/2011 9:19:33 AM by FX (modified after loading)		
Analysis Method	: D:\LC\201110\FX\FX-4-81\FX-4-81 2011-11-01 09-02-33\021-0101.D\DA.M (ADH-10-90-1ML-254NM.M)		
Last changed	: 11/1/2011 12:29:50 PM by LTL (modified after loading)		



Area Percent Report

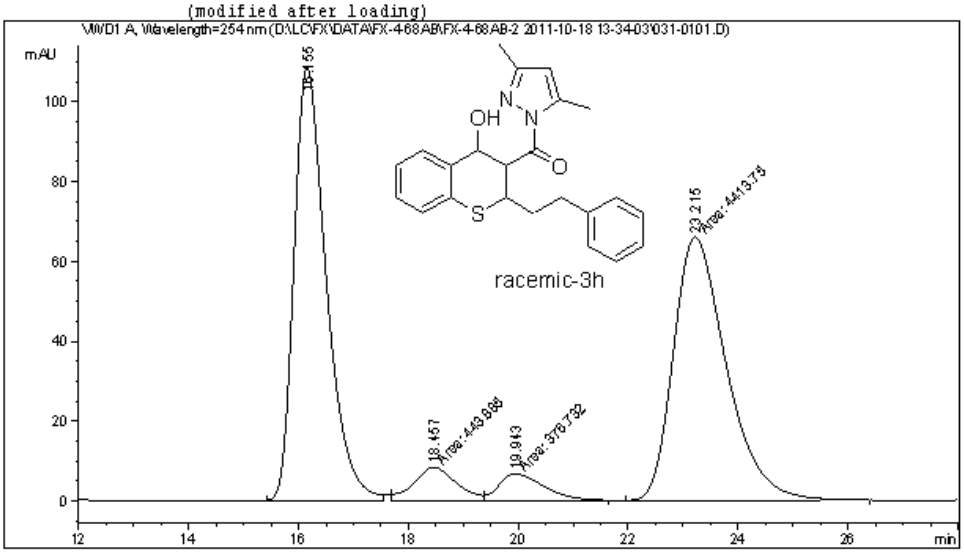
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	5.848	MM	0.2234	18.27868	1.36362	0.8898
2	7.039	MF	0.2488	25.14719	1.68490	1.2242
3	7.716	FM	0.2951	2000.30615	112.95503	97.3788
4	9.295	MM	0.3111	10.41851	5.58161e-1	0.5072
Totals :				2054.15053	116.56172	

Data File D:\LC\FX\DATA\FX-4-68AB\FX-4-68AB-2 2011-10-18 13-34-03\031-0101.D
Sample Name: FX-4-68Ab

Acq. Operator	: FX	Seq. Line	: 1
Acq. Instrument	: Instrument 1	Location	: Vial 31
Injection Date	: 10/18/2011 1:35:19 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\FX\Data\FX-4-68AB\FX-4-68AB-2 2011-10-18 13-34-03\ASH-10-90-05ML-254NM.M		
Last changed	: 9/27/2011 4:31:55 PM by FX		
Analysis Method	: D:\LC\FX\DATA\FX-4-68AB\FX-4-68AB-2 2011-10-18 13-34-03\031-0101.D\DA.M (ASH-10-90-05ML-254NM.M)		
Last changed	: 10/18/2011 3:04:21 PM by FX (modified after loading)		



Area Percent Report

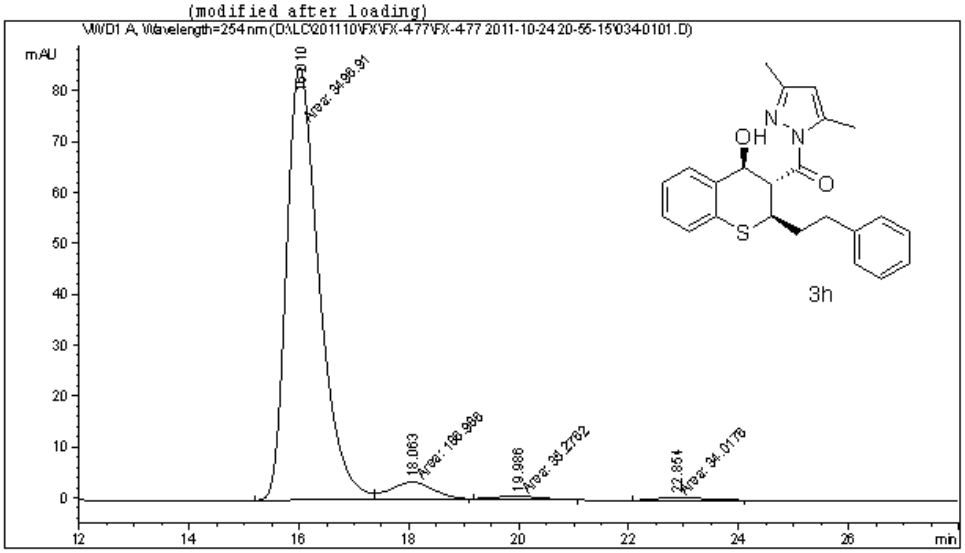
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	16.155	BB	0.6155	4413.90527	108.58633	45.7482
2	18.457	FM	0.8915	443.88470	8.29829	4.6007
3	19.943	FM	0.9546	376.73221	6.57743	3.9047
4	23.215	MM	1.1149	4413.74658	65.98317	45.7465
Totals :				9648.26877	189.44522	

Data File D:\LC\201110\FX\FX-4-77\FX-4-77 2011-10-24 20-55-15\034-0101.D
Sample Name: FX-4-77Ab

Acq. Operator	: FX	Seq. Line	: 1
Acq. Instrument	: Instrument 1	Location	: Vial 34
Injection Date	: 10/24/2011 8:56:34 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201110\FX\FX-4-77\FX-4-77 2011-10-24 20-55-15\ASH-10-90-05ML-254NM-30MIN.M		
Last changed	: 9/28/2011 11:47:06 AM by thl		
Analysis Method	: D:\LC\201110\FX\FX-4-77\FX-4-77 2011-10-24 20-55-15\034-0101.D\DA.M (ASH-10-90-05ML-254NM-30MIN.M)		
Last changed	: 10/25/2011 1:44:07 PM by FX (modified after loading)		



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	16.010	MF	0.6851	3496.90649	85.07250	93.1717
2	18.063	FM	0.9061	186.98566	3.43938	4.9821
3	19.986	MM	1.0157	35.27618	5.78854e-1	0.9399
4	22.854	MM	1.0103	34.01763	5.61163e-1	0.9064
Totals :				3753.18596	89.65190	

```
Data File D:\LC\201110\FX\FX-4-78B\FX-4-78B 2011-10-25 11-55-52\025-0101.D
Sample Name: FX-4-78b

    ==================================================================
    Acq. Operator   : hzl                          Seq. Line :   1
    Acq. Instrument : Instrument 1                  Location : Vial 25
    Injection Date  : 10/25/2011 11:57:30 AM             Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\201110\FX\FX-4-78B\FX-4-78B 2011-10-25 11-55-52\0JH-10-90-1ML-254NM.
                      M
    Last changed    : 10/25/2011 12:12:48 PM by hzl
                      (modified after loading)
    Analysis Method : D:\LC\201110\FX\FX-4-78B\FX-4-78B 2011-10-25 11-55-52\025-0101.D\DA.M (
                      0JH-10-90-1ML-254NM.M)
    Last changed    : 10/25/2011 1:49:22 PM by FX
                      (modified after loading)
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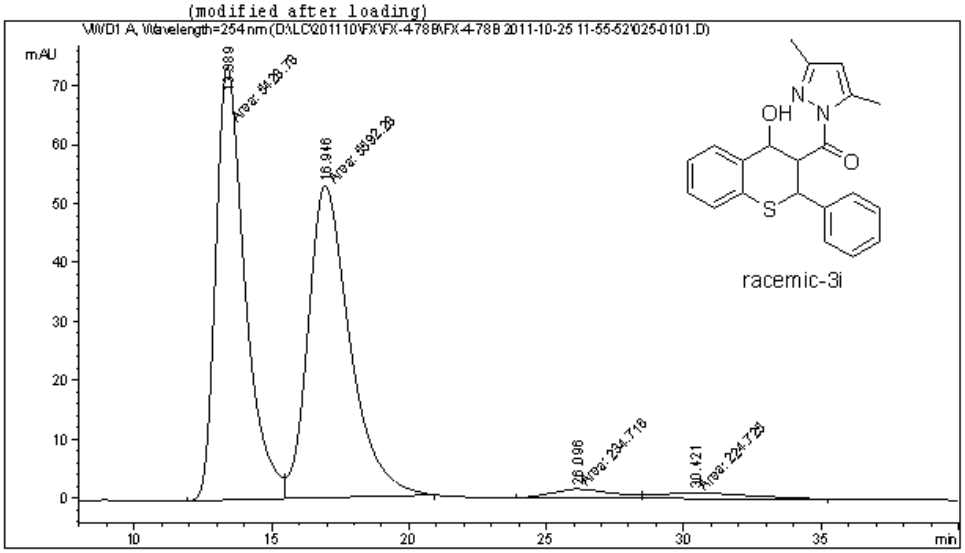



```
    ==================================================================
                          Area Percent Report
    ==================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=254 nm

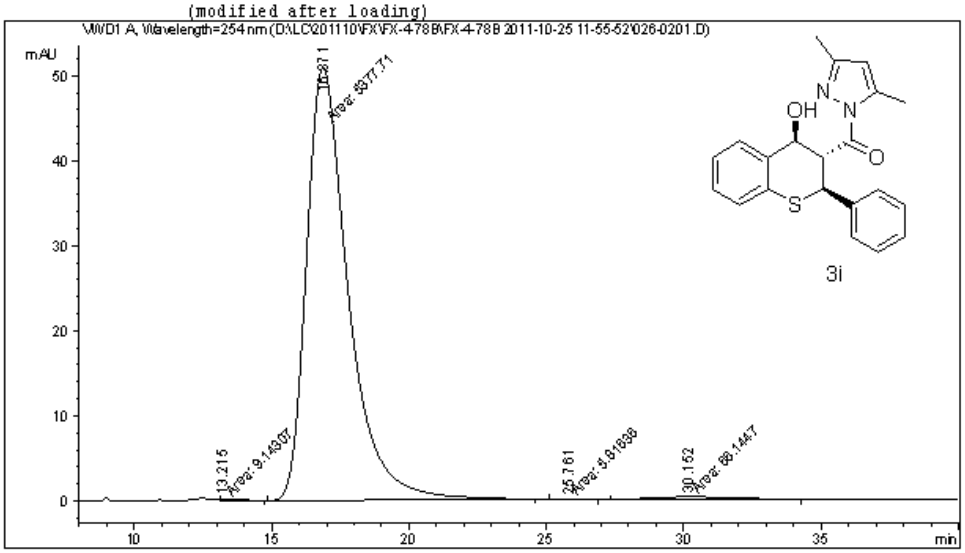
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  13.389 MF    1.2321 5428.77588   73.43800  47.2869
       2  16.946 FM    1.7612 5592.28076   52.92031  48.7111
       3  26.096 MF    2.5482  234.71635    1.53520   2.0445
       4  30.421 FM    3.8967  224.72464 9.61171e-1   1.9574

    Totals :                  1.14805e4   128.85469

Instrument 1 10/25/2011 1:49:26 PM FX                          Page 1 of 2
```

Data File D:\LC\201110\FX\FX-4-78B\FX-4-78B 2011-10-25 11-55-52\026-0201.D
Sample Name: FX-4-77Cb

Acq. Operator	: hzl	Seq. Line	: 2
Acq. Instrument	: Instrument 1	Location	: Vial 26
Injection Date	: 10/25/2011 12:58:45 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201110\FX\FX-4-78B\FX-4-78B 2011-10-25 11-55-52\0JH-10-90-1ML-254NM.M		
Last changed	: 10/25/2011 1:46:30 PM by hzl (modified after loading)		
Analysis Method	: D:\LC\201110\FX\FX-4-78B\FX-4-78B 2011-10-25 11-55-52\026-0201.D\DA.M (0JH-10-90-1ML-254NM.M)		
Last changed	: 10/25/2011 2:03:36 PM by FX (modified after loading)		



Area Percent Report

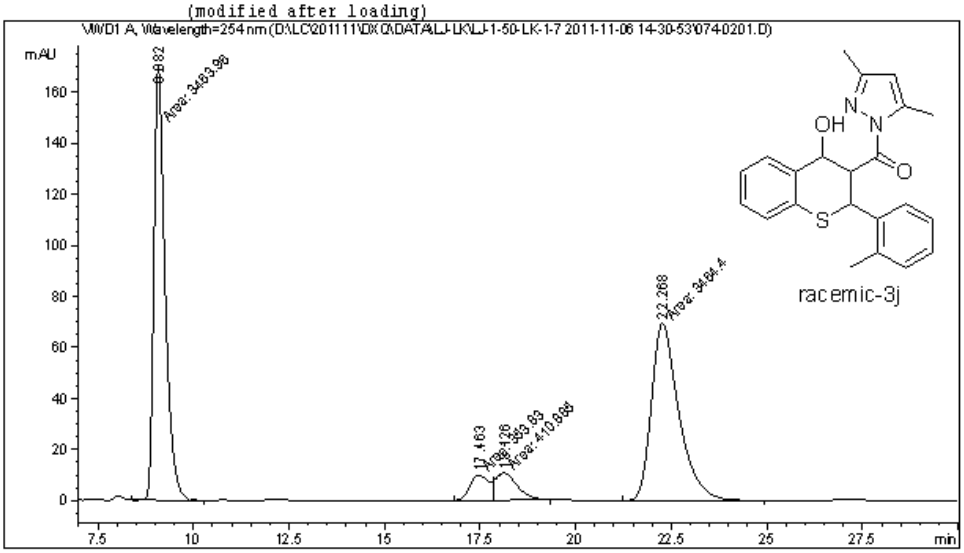
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	13.215	MM	0.8607	9.14307	1.77046e-1	0.1675
2	16.871	MM	1.7618	5377.70947	50.87416	98.5143
3	25.761	MM	1.0419	5.81636	9.30364e-2	0.1065
4	30.152	MM	3.5486	66.14468	3.10659e-1	1.2117
Totals :				5458.81357	51.45490	

Data File D:\LC\201111\DXQ\DATA\LJ-LK\LJ-1-50-LK-1-7 2011-11-06 14-30-53\074-0201.D
Sample Name: lk-1-7a

Acq. Operator	: thl	Seq. Line	: 2
Acq. Instrument	: Instrument 1	Location	: Vial 74
Injection Date	: 11/6/2011 3:34:05 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201111\dxq\data\LJ-LK\LJ-1-50-LK-1-7 2011-11-06 14-30-53\ADH-10-90-1ML-254NM-60MIN.M		
Last changed	: 11/1/2011 5:36:20 PM by LTL		
Analysis Method	: D:\LC\201111\DXQ\DATA\LJ-LK\LJ-1-50-LK-1-7 2011-11-06 14-30-53\074-0201.D\DA.M (ADH-10-90-1ML-254NM-60MIN.M)		
Last changed	: 11/10/2011 8:48:14 PM by THL (modified after loading)		



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

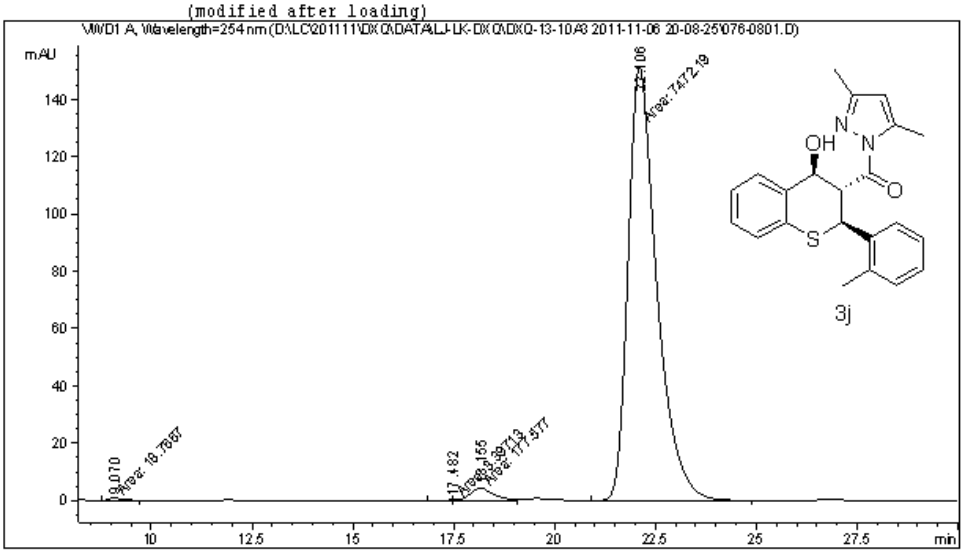
Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	9.082	MM	0.3402	3463.95972	169.68002	45.0270
2	17.463	MF	0.5906	353.83002	9.98481	4.5993
3	18.126	FM	0.6422	410.88525	10.66342	5.3410
4	22.268	MM	0.8316	3464.39648	69.43513	45.0327
Totals :				7693.07147	259.76338	

Instrument 1 11/10/2011 8:48:56 PM THL

Data File D:\LC\201111\DXQ\DATA\LJ-LK-DXQ\DXQ-13-10A3 2011-11-06 20-08-25\076-0801.D
Sample Name: lk-1-7c

Acq. Operator	: thl	Seq. Line	: 8
Acq. Instrument	: Instrument 1	Location	: Vial 76
Injection Date	: 11/7/2011 12:45:09 AM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201111\dxq\data\LJ-LK-DXQ\DXQ-13-10A3 2011-11-06 20-08-25\ADH-10-90-1ML-254NM-30MIN.M		
Last changed	: 11/5/2011 7:55:50 PM by TMC		
Analysis Method	: D:\LC\201111\DXQ\DATA\LJ-LK-DXQ\DXQ-13-10A3 2011-11-06 20-08-25\076-0801.D\DA.M (ADH-10-90-1ML-254NM-30MIN.M)		
Last changed	: 11/10/2011 9:04:08 PM by THL (modified after loading)		



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

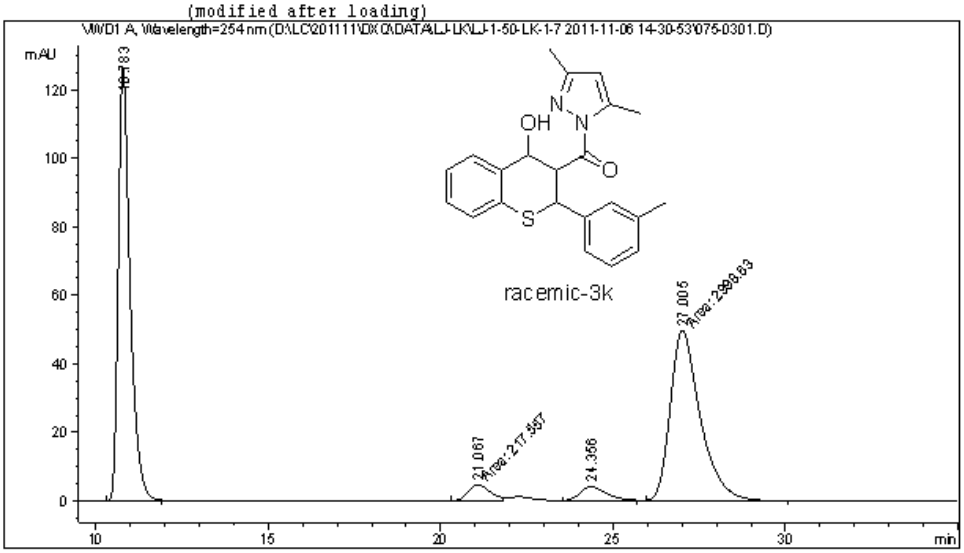
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	9.070	MM	0.3406	18.76665	9.18196e-1	0.2446
2	17.482	MF	0.3135	3.39713	1.80607e-1	0.0443
3	18.155	FM	0.6816	177.57684	4.34195	2.3146
4	22.106	MM	0.8239	7472.19238	151.15479	97.3965
Totals :				7671.93301	156.59554	

Instrument 1 11/10/2011 9:04:22 PM THL

Page 1 of 2

Data File D:\LC\201111\DXQ\DATA\LJ-LK\LJ-1-50-LK-1-7 2011-11-06 14-30-53\075-0301.D
Sample Name: lk-1-7b

Acq. Operator	: thl	Seq. Line	: 3
Acq. Instrument	: Instrument 1	Location	: Vial 75
Injection Date	: 11/6/2011 4:35:24 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201111\dxq\data\LJ-LK\LJ-1-50-LK-1-7 2011-11-06 14-30-53\ADH-10-90-1ML-254NM-60MIN.M		
Last changed	: 11/6/2011 5:11:35 PM by thl (modified after loading)		
Analysis Method	: D:\LC\201111\DXQ\DATA\LJ-LK\LJ-1-50-LK-1-7 2011-11-06 14-30-53\075-0301.D\DA.M (ADH-10-90-1ML-254NM-60MIN.M)		
Last changed	: 11/10/2011 8:51:33 PM by THL (modified after loading)		



Area Percent Report

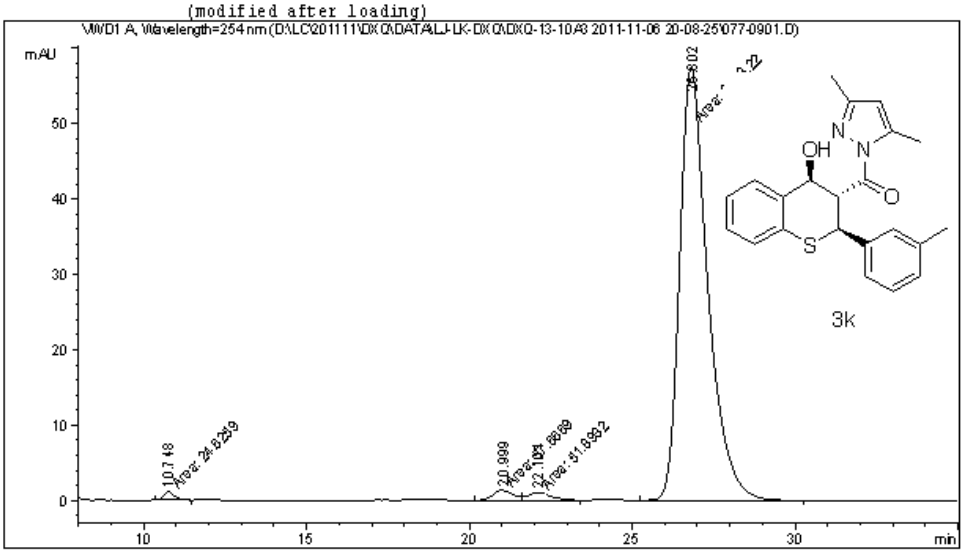
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	10.783	BB	0.3547	3008.43262	126.48126	46.7640
2	21.067	MM	0.7546	217.55737	4.80504	3.3818
3	24.356	BB	0.7460	208.61406	4.03435	3.2428
4	27.005	MM	1.0065	2998.62500	49.65536	46.6115
Totals :				6433.22905	184.97601	

Data File D:\LC\2011l1\DXQ\DATA\LJ-LK-DXQ\DXQ-13-10A3 2011-11-06 20-08-25\077-0901.D
Sample Name: lk-1-7d

Acq. Operator	: thl	Seq. Line	: 9
Acq. Instrument	: Instrument 1	Location	: Vial 77
Injection Date	: 11/7/2011 1:16:35 AM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\2011l1\dxq\data\LJ-LK-DXQ\DXQ-13-10A3 2011-11-06 20-08-25\ADH-10-90-1ML-254NM-40MIN.M		
Last changed	: 11/1/2011 4:42:27 PM by LTL		
Analysis Method	: D:\LC\2011l1\DXQ\DATA\LJ-LK-DXQ\DXQ-13-10A3 2011-11-06 20-08-25\077-0901.D\DA.M (ADH-10-90-1ML-254NM-40MIN.M)		
Last changed	: 11/10/2011 9:08:21 PM by THL (modified after loading)		



Area Percent Report

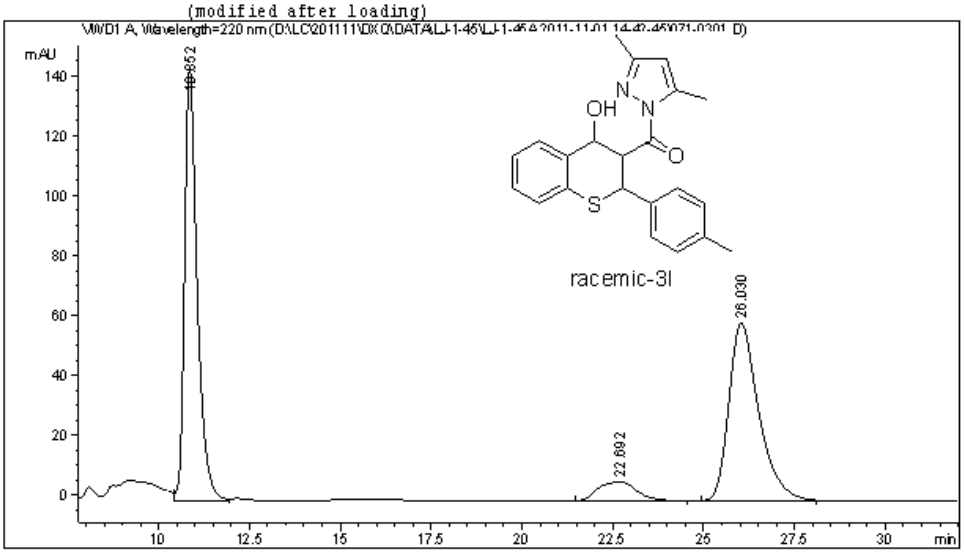
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	10.748	MM	0.3781	24.62591	1.08547	0.6865
2	20.999	MF	0.7144	57.66687	1.34530	1.6075
3	22.101	FM	0.8278	51.89318	1.04475	1.4465
4	26.802	MM	1.0020	3453.22095	57.43696	96.2595
Totals :				3587.40691	60.91248	

Data File D:\LC\201111\DXQ\DATA\LJ-1-45\LJ-1-45A 2011-11-01 14-42-45\071-0301.D
Sample Name: LJ-1-45A

Acq. Operator	: LTL	Seq. Line	: 3
Acq. Instrument	: Instrument 1	Location	: Vial 71
Injection Date	: 11/1/2011 3:57:09 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201111\dxq\data\LJ-1-45\LJ-1-45A 2011-11-01 14-42-45\ADH-10-90-1ML-220.M		
Last changed	: 11/1/2011 4:33:51 PM by LTL (modified after loading)		
Analysis Method	: D:\LC\201111\DXQ\DATA\LJ-1-45\LJ-1-45A 2011-11-01 14-42-45\071-0301.D\DA.M (ADH-10-90-1ML-220.M)		
Last changed	: 1/1/2012 5:59:29 PM by thl (modified after loading)		



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

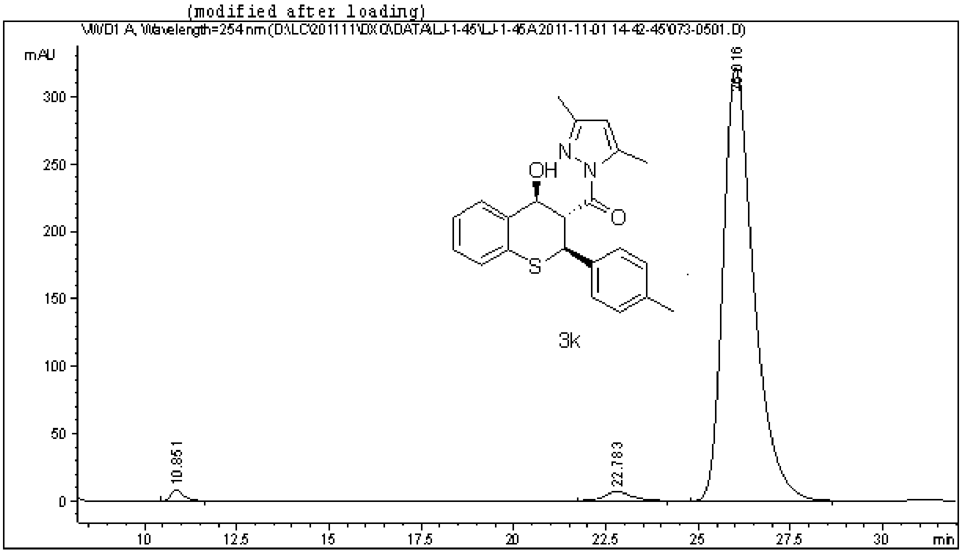
Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	10.852	VB	0.3596	3478.65356	144.45667	47.4977
2	22.692	BB	0.9464	460.54907	6.44053	6.2884
3	26.030	BB	0.8566	3384.63452	59.29886	46.2140
Totals :				7323.83716	210.19606	

Data File D:\LC\201111\DXQ\DATA\LJ-1-45\LJ-1-45A 2011-11-01 14-42-45\073-0501.D
Sample Name: LJ-1-45C

===

Acq. Operator : LTL   Seq. Line : 5
Acq. Instrument : Instrument 1   Location : Vial 73
Injection Date : 11/1/2011 5:29:11 PM   Inj : 1
Inj Volume : 5 µl
Acq. Method : D:\LC\201111\dxq\data\LJ-1-45\LJ-1-45A 2011-11-01 14-42-45\ADH-10-90-1ML-254NM-40MIN.M
Last changed : 11/1/2011 4:42:27 PM by LTL
Analysis Method : D:\LC\201111\DXQ\DATA\LJ-1-45\LJ-1-45A 2011-11-01 14-42-45\073-0501.D\DA.M (ADH-10-90-1ML-254NM-40MIN.M)
Last changed : 1/1/2012 6:00:34 PM by thl
(modified after loading)



===

Area Percent Report

===

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	10.851	BB	0.3511	193.66840	8.20399	1.0147
2	22.783	BB	0.7574	367.73703	6.99643	1.9268
3	26.016	BB	0.8628	1.85241e4	321.58984	97.0585
Totals :				1.90855e4	336.79026	

Instrument 1 1/1/2012 6:00:39 PM thl   Page 1 of 2

Data File D:\LC\201111\DXQ\DATA\LJ-1-45\LJ-1-45A 2011-11-01 14-42-45\072-0401.D
Sample Name: LJ-1-45B

```
==================================================================
Acq. Operator   : LTL                          Seq. Line :   4
Acq. Instrument : Instrument 1                  Location : Vial 72
Injection Date  : 11/1/2011 4:38:45 PM               Inj :   1
                                               Inj Volume : 5 µl
Acq. Method     : D:\LC\201111\dxq\data\LJ-1-45\LJ-1-45A 2011-11-01 14-42-45\ADH-10-90-1ML-
                  220.M
Last changed    : 11/1/2011 5:27:25 PM by LTL
                  (modified after loading)
Analysis Method : D:\LC\201111\DXQ\DATA\LJ-1-45\LJ-1-45A 2011-11-01 14-42-45\072-0401.D\DA.M
                  (ADH-10-90-1ML-220.M)
Last changed    : 11/10/2011 7:20:24 PM by THL
                  (modified after loading)
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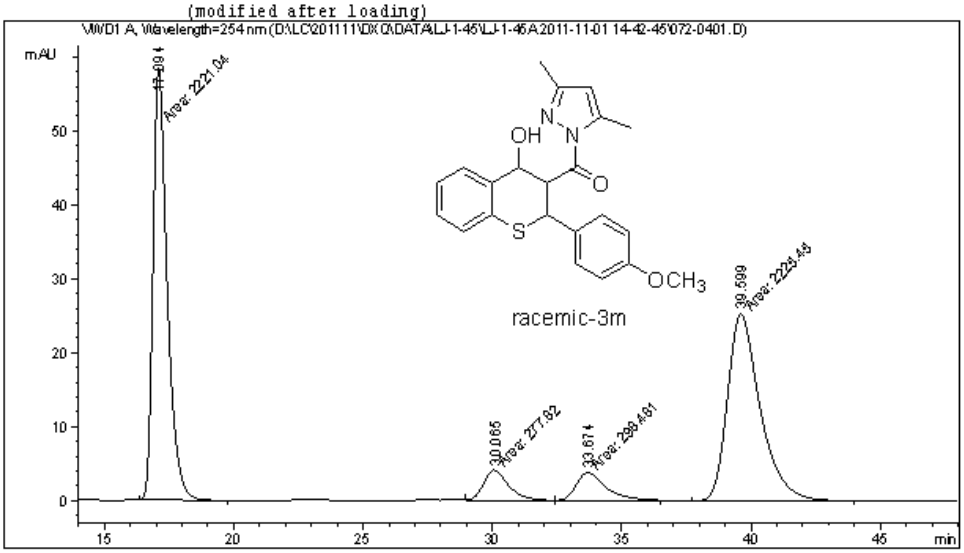
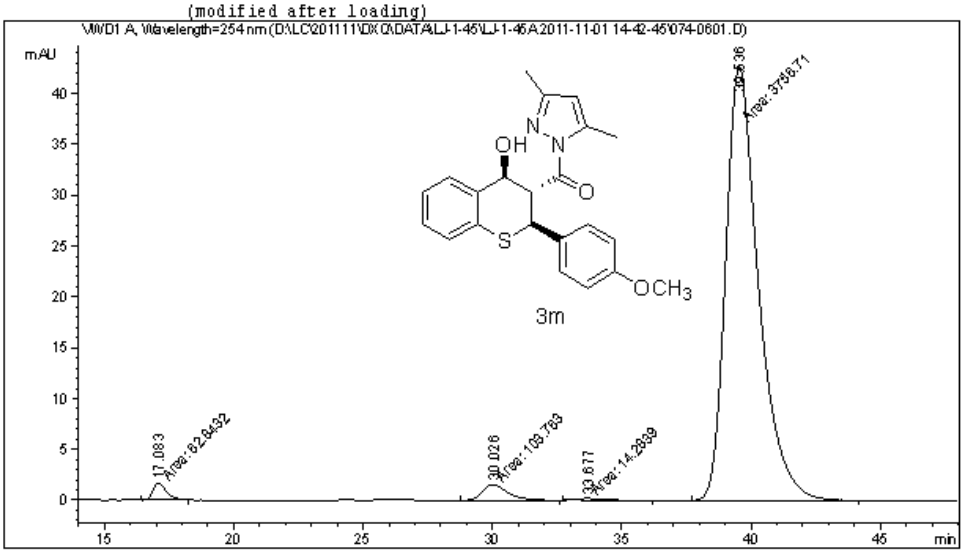



```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  17.094 MM    0.6327 2221.04443   58.51120  44.2193
   2  30.065 MM    1.1256  277.81979    4.11376   5.5312
   3  33.674 MM    1.3181  298.48105    3.77402   5.9425
   4  39.599 MM    1.4658 2225.44946   25.30422  44.3070

Totals :                  5022.79474   91.70320
```

Data File D:\LC\201111\DXQ\DATA\LJ-1-45\LJ-1-45A 2011-11-01 14-42-45\074-0601.D
Sample Name: LJ-1-45D

Acq. Operator	: LTL	Seq. Line	: 6
Acq. Instrument	: Instrument 1	Location	: Vial 74
Injection Date	: 11/1/2011 6:10:57 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201111\dxq\data\LJ-1-45\LJ-1-45A 2011-11-01 14-42-45\ADH-10-90-1ML-254NM-60MIN.M		
Last changed	: 11/1/2011 5:36:20 PM by LTL		
Analysis Method	: D:\LC\201111\DXQ\DATA\LJ-1-45\LJ-1-45A 2011-11-01 14-42-45\074-0601.D\DA.M (ADH-10-90-1ML-254NM-60MIN.M)		
Last changed	: 11/10/2011 7:36:15 PM by THL (modified after loading)		



Area Percent Report

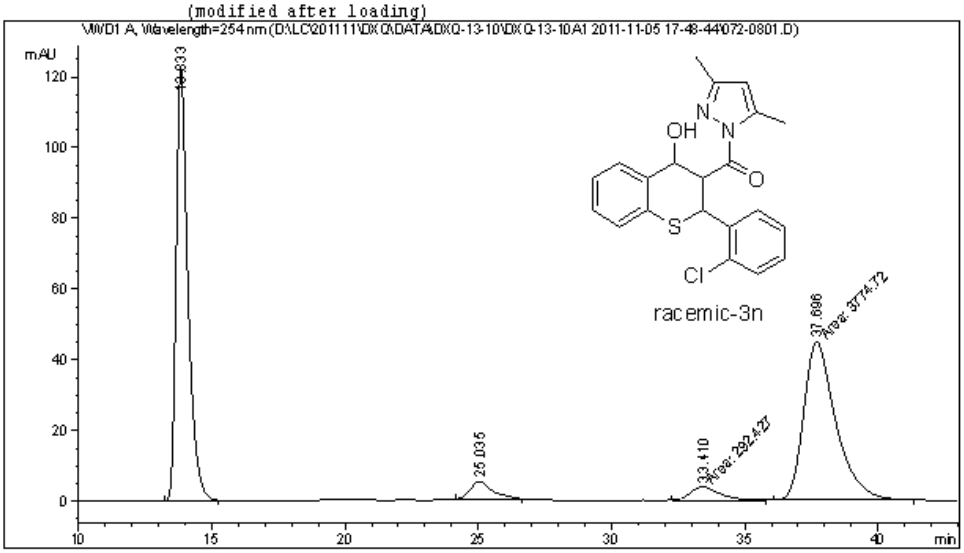
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	17.083	MM	0.6247	62.84315	1.67652	1.5960
2	30.026	MM	1.1155	103.76342	1.55036	2.6352
3	33.677	MM	1.2817	14.28390	1.85747e-1	0.3628
4	39.536	MM	1.4687	3756.70654	42.63120	95.4061
Totals :				3937.59702	46.04383	

Data File D:\LC\201111\DXQ\DATA\DXQ-13-10\DXQ-13-10A1 2011-11-05 17-48-44\072-0801.D
Sample Name: lj-1-50a

Acq. Operator	: TMC	Seq. Line	: 8
Acq. Instrument	: Instrument 1	Location	: Vial 72
Injection Date	: 11/5/2011 9:32:55 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201111\dxq\data\DXQ-13-10\DXQ-13-10A1 2011-11-05 17-48-44\ADH-10-90-1ML-254NM-30MIN.M		
Last changed	: 11/5/2011 10:15:53 PM by TMC (modified after loading)		
Analysis Method	: D:\LC\201111\DXQ\DATA\DXQ-13-10\DXQ-13-10A1 2011-11-05 17-48-44\072-0801.D\DA.M (ADH-10-90-1ML-254NM-30MIN.M)		
Last changed	: 11/10/2011 8:19:34 PM by THL (modified after loading)		



Area Percent Report

Sorted By	:	Signal	
Multiplier	:	1.0000	
Dilution	:	1.0000	

Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

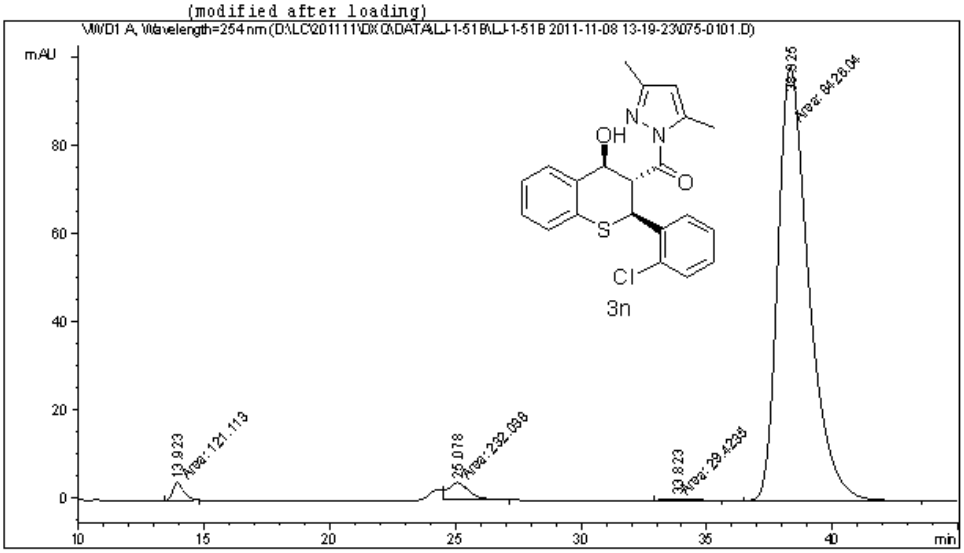
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	13.833	BB	0.4564	3754.91797	122.55990	46.2695
2	25.035	BB	0.8282	293.25409	4.96974	3.6136
3	33.410	MM	1.2733	292.42661	3.82775	3.6034
4	37.696	MM	1.4035	3774.72168	44.82587	46.5135
Totals :				8115.32034	176.18326	

Instrument 1 11/10/2011 8:19:39 PM THL

Page 1 of 2

Data File D:\LC\201111\DXQ\DATA\LJ-1-51B\LJ-1-51B 2011-11-08 13-19-23\075-0101.D
Sample Name: lj-1-51b

Acq. Operator	: THL	Seq. Line	: 1
Acq. Instrument	: Instrument 1	Location	: Vial 75
Injection Date	: 11/8/2011 1:20:46 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201111\dxq\data\LJ-1-51B\LJ-1-51B 2011-11-08 13-19-23\ADH-10-90-1ML-254NM-45MIN.M		
Last changed	: 11/6/2011 7:43:25 PM by thl		
Analysis Method	: D:\LC\201111\DXQ\DATA\LJ-1-51B\LJ-1-51B 2011-11-08 13-19-23\075-0101.D\DA.M (ADH-10-90-1ML-254NM-45MIN.M)		
Last changed	: 11/10/2011 8:27:28 PM by THL (modified after loading)		



Area Percent Report

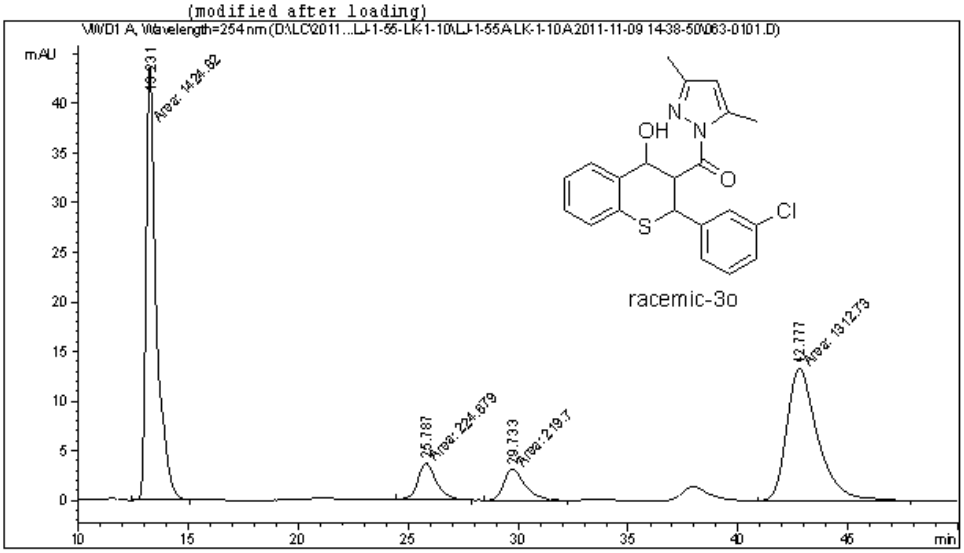
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	13.923	MM	0.4983	121.11275	4.05103	1.3749
2	25.078	MM	0.9827	232.03789	3.93548	2.6342
3	33.823	MM	1.1720	29.42353	4.18433e-1	0.3340
4	38.325	MM	1.4250	8426.04004	98.55006	95.6568
Totals :				8808.61421	106.95501	

Data File D:\LC\2011...TA\LJ-1-55-LK-1-10\LJ-1-55A-LK-1-10A 2011-11-09 14-38-50\063-0101.D
Sample Name: LJ-1-55A

Acq. Operator	: THL	Seq. Line	: 1
Acq. Instrument	: Instrument 1	Location	: Vial 63
Injection Date	: 11/9/2011 2:40:52 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\2011l1\dxq\data\LJ-1-55-LK-1-10\LJ-1-55A-LK-1-10A 2011-11-09 14-38-50\ADH-10-90-1ML-254NM-40MIN.M		
Last changed	: 11/9/2011 3:34:56 PM by THL (modified after loading)		
Analysis Method	: D:\LC\2011l1\DXQ\DATA\LJ-1-55-LK-1-10\LJ-1-55A-LK-1-10A 2011-11-09 14-38-50\063-0101.D\DA.M (ADH-10-90-1ML-254NM-40MIN.M)		
Last changed	: 11/10/2011 8:34:43 PM by THL (modified after loading)		



Area Percent Report

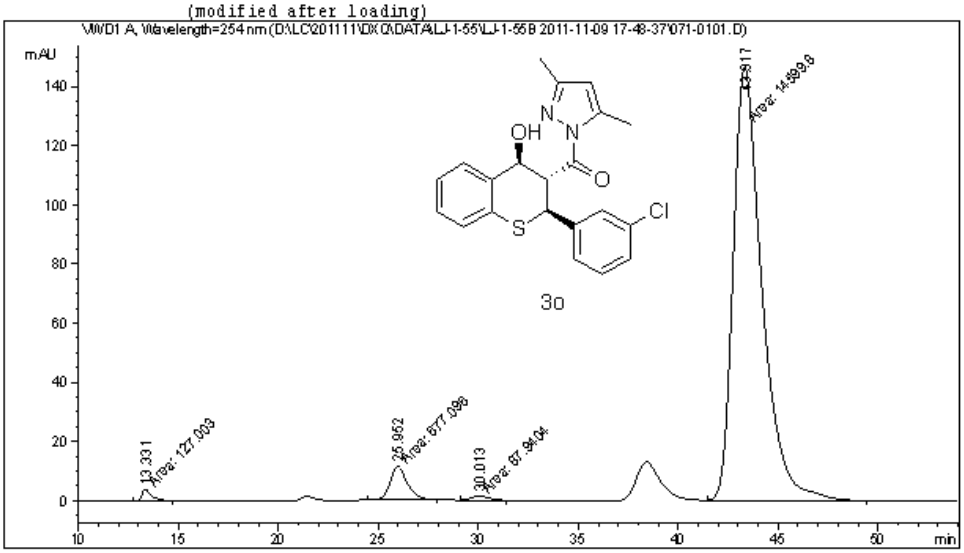
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	13.231	MM	0.5447	1424.82349	43.59535	44.7786
2	25.787	MM	1.0076	224.67863	3.71627	7.0611
3	29.733	MM	1.1467	219.69971	3.19326	6.9046
4	42.777	MM	1.6416	1312.72913	13.32735	41.2557
Totals :				3181.93095	63.83223	

Data File D:\LC\201111\DXQ\DATA\LJ-1-55\LJ-1-55B 2011-11-09 17-48-37\071-0101.D
Sample Name: LJ-1-55B

Acq. Operator	: THL	Seq. Line	: 1
Acq. Instrument	: Instrument 1	Location	: Vial 71
Injection Date	: 11/9/2011 5:49:59 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201111\dxq\data\LJ-1-55\LJ-1-55B 2011-11-09 17-48-37\ADH-10-90-1ML-254NM-60MIN.M		
Last changed	: 11/9/2011 6:42:24 PM by THL (modified after loading)		
Analysis Method	: D:\LC\201111\DXQ\DATA\LJ-1-55\LJ-1-55B 2011-11-09 17-48-37\071-0101.D\DA.M (ADH-10-90-1ML-254NM-60MIN.M)		
Last changed	: 11/10/2011 8:37:43 PM by THL (modified after loading)		



Area Percent Report

Sorted By	:	Signal	
Multiplier	:	1.0000	
Dilution	:	1.0000	

Use Multiplier & Dilution Factor with ISTDs

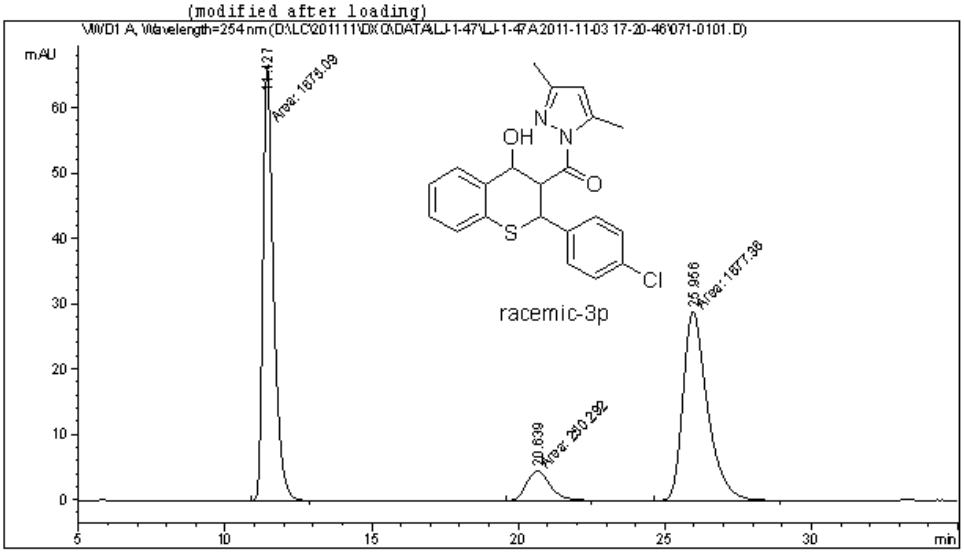
Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU*s	Height [mAU]	Area %
1	13.331	MM	0.5474	127.00271	3.86668	0.8198
2	25.952	MM	0.9905	677.09619	11.39303	4.3707
3	30.013	MM	1.0066	87.94037	1.45608	0.5677
4	43.317	MM	1.6637	1.45998e4	146.25407	94.2419
Totals :				1.54918e4	162.96986	

Instrument 1 11/10/2011 8:37:49 PM THL

Data File D:\LC\201111\DXQ\DATA\LJ-1-47\LJ-1-47A 2011-11-03 17-20-46\071-0101.D
Sample Name: LJ-1-47A

Acq. Operator	: LTL	Seq. Line	: 1
Acq. Instrument	: Instrument 1	Location	: Vial 71
Injection Date	: 11/3/2011 5:22:16 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201111\dxq\data\LJ-1-47\LJ-1-47A 2011-11-03 17-20-46\ADH-10-90-1ML-254NM.M		
Last changed	: 11/3/2011 6:13:11 PM by LTL (modified after loading)		
Analysis Method	: D:\LC\201111\DXQ\DATA\LJ-1-47\LJ-1-47A 2011-11-03 17-20-46\071-0101.D\DA.M (ADH-10-90-1ML-254NM.M)		
Last changed	: 11/10/2011 7:38:54 PM by THL (modified after loading)		



Area Percent Report

Sorted By	:	Signal				
Multiplier	:	1.0000				
Dilution	:	1.0000				

Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU*s	Height [mAU]	Area %
1	11.427	MM	0.4210	1675.08911	66.31800	46.4949
2	20.639	MM	0.9280	250.29192	4.49513	6.9473
3	25.956	MM	0.9664	1677.35852	28.92724	46.5579
Totals :				3602.73955	99.74037	

Data File D:\LC\201111\DXQ\DATA\LJ-1-47\LJ-1-47A 2011-11-03 19-09-06\073-0101.D
Sample Name: LJ-1-47C

```
==================================================================
Acq. Operator   : LTL                          Seq. Line :   1
Acq. Instrument : Instrument 1                  Location : Vial 73
Injection Date  : 11/3/2011 7:10:21 PM             Inj :   1
                                                Inj Volume : 5 µl
Acq. Method     : D:\LC\201111\dxq\data\LJ-1-47\LJ-1-47A 2011-11-03 19-09-06\ADH-10-90-1ML-
                  254NM.M
Last changed    : 11/3/2011 7:10:58 PM by LTL
                  (modified after loading)
Analysis Method : D:\LC\201111\DXQ\DATA\LJ-1-47\LJ-1-47A 2011-11-03 19-09-06\073-0101.D\DA.M
                  (ADH-10-90-1ML-254NM.M)
Last changed    : 11/10/2011 7:57:07 PM by THL
                  (modified after loading)
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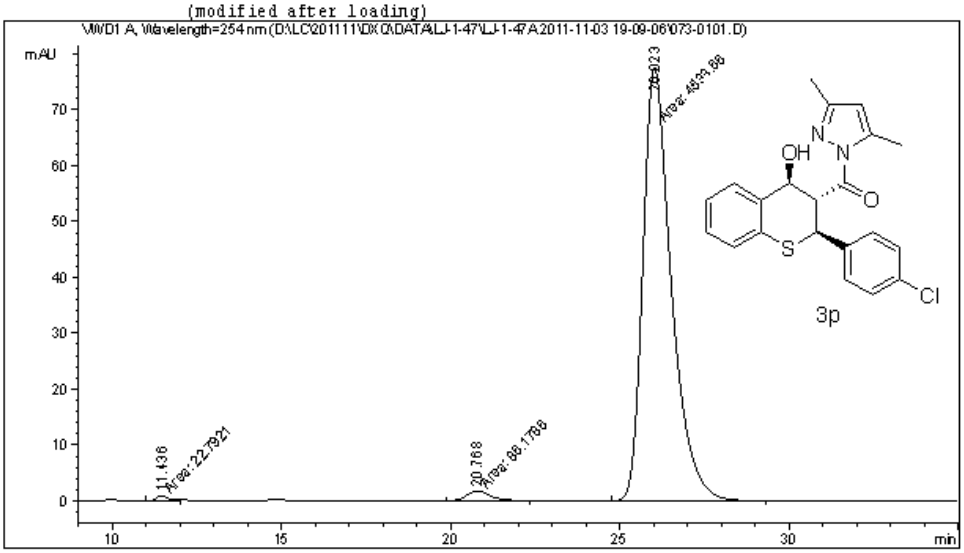



```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs

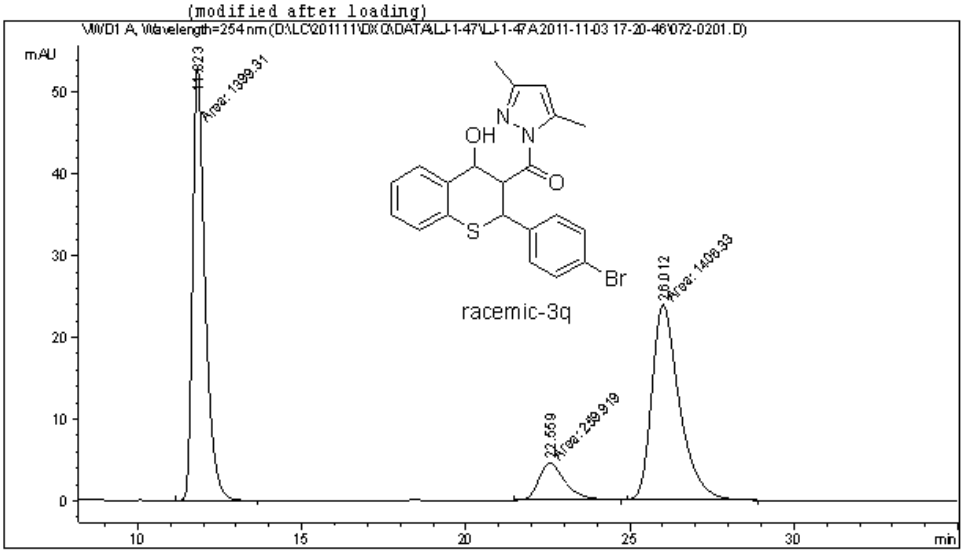

Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  11.436 MM    0.4284   22.79212 8.86666e-1   0.4909
   2  20.768 MM    0.8196   86.17857    1.75242   1.8562
   3  26.023 MM    0.9745 4533.66162   77.54041  97.6528

Totals :                  4642.63231   80.17950
```

Data File D:\LC\201111\DXQ\DATA\LJ-1-47\LJ-1-47A 2011-11-03 17-20-46\072-0201.D
Sample Name: LJ-1-47B

Acq. Operator	: LTL	Seq. Line	: 2
Acq. Instrument	: Instrument 1	Location	: Vial 72
Injection Date	: 11/3/2011 6:15:09 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201111\dxq\data\LJ-1-47\LJ-1-47A 2011-11-03 17-20-46\ADH-10-90-1ML-254NM.M		
Last changed	: 11/3/2011 6:13:46 PM by LTL (modified after loading)		
Analysis Method	: D:\LC\201111\DXQ\DATA\LJ-1-47\LJ-1-47A 2011-11-03 17-20-46\072-0201.D\DA.M (ADH-10-90-1ML-254NM.M)		
Last changed	: 11/10/2011 7:43:54 PM by THL (modified after loading)		



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	11.823	MM	0.4400	1399.31262	53.00880	45.6463
2	22.559	MM	0.9469	259.91943	4.57490	8.4787
3	26.012	MM	0.9800	1406.32507	23.91792	45.8750
Totals :				3065.55713	81.50162	

Data File D:\LC\201111\DXQ\DATA\LJ-1-47\LJ-1-47A 2011-11-03 19-09-06\074-0201.D
Sample Name: LJ-1-47D

```
==================================================================
Acq. Operator   : LTL                          Seq. Line :   2
Acq. Instrument : Instrument 1                  Location : Vial 74
Injection Date  : 11/3/2011 7:46:57 PM             Inj :   1
                                                Inj Volume : 5 µl
Acq. Method     : D:\LC\201111\dxq\data\LJ-1-47\LJ-1-47A 2011-11-03 19-09-06\ADH-10-90-1ML-
                  254NM.M
Last changed    : 11/3/2011 8:21:37 PM by LTL
                  (modified after loading)
Analysis Method : D:\LC\201111\DXQ\DATA\LJ-1-47\LJ-1-47A 2011-11-03 19-09-06\074-0201.D\DA.M
                  (ADH-10-90-1ML-254NM.M)
Last changed    : 11/10/2011 8:01:00 PM by THL
                  (modified after loading)
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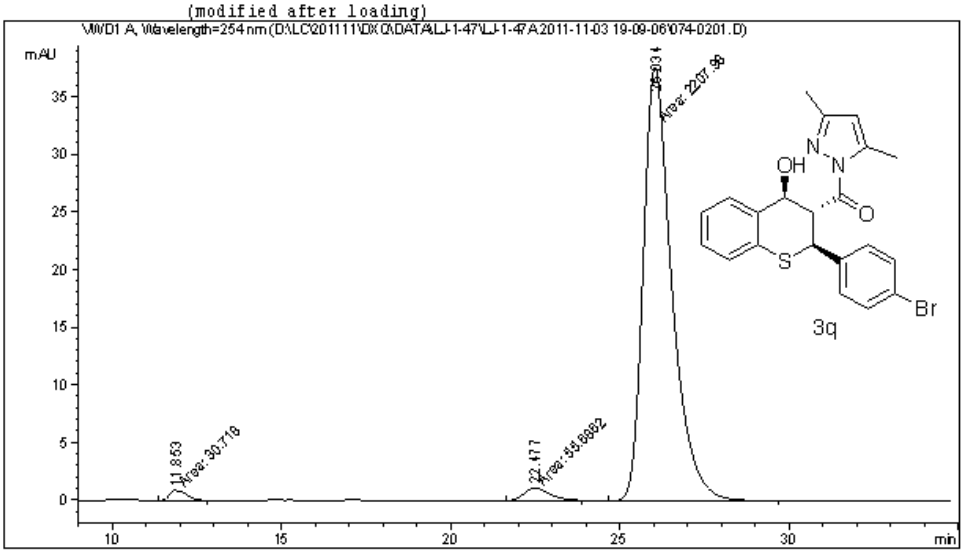


```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  11.853 MM    0.5825   30.71800 8.78972e-1   1.3388
   2  22.477 MM    0.8501   55.68617    1.09181   2.4271
   3  26.034 MM    0.9788 2207.97803   37.59824  96.2341

Totals :                  2294.38220   39.56902
```

Data File D:\LC\201111\DXQ\DATA\LJ-1-57\LJ-1-57A1 2011-11-10 15-02-42\071-0101.D
Sample Name: LJ-1-57A

```
==================================================================
Acq. Operator   : THL                          Seq. Line :   1
Acq. Instrument : Instrument 1                  Location : Vial 71
Injection Date  : 11/10/2011 3:03:56 PM              Inj :   1
                                               Inj Volume : 5 µl
Acq. Method     : D:\LC\201111\dxq\data\LJ-1-57\LJ-1-57A1 2011-11-10 15-02-42\ADH-10-90-1ML-
                  254NM.M
Last changed    : 11/10/2011 3:58:02 PM by THL
                  (modified after loading)
Analysis Method : D:\LC\201111\DXQ\DATA\LJ-1-57\LJ-1-57A1 2011-11-10 15-02-42\071-0101.D\DA.
                  M (ADH-10-90-1ML-254NM.M)
Last changed    : 11/10/2011 8:40:44 PM by THL
                  (modified after loading)
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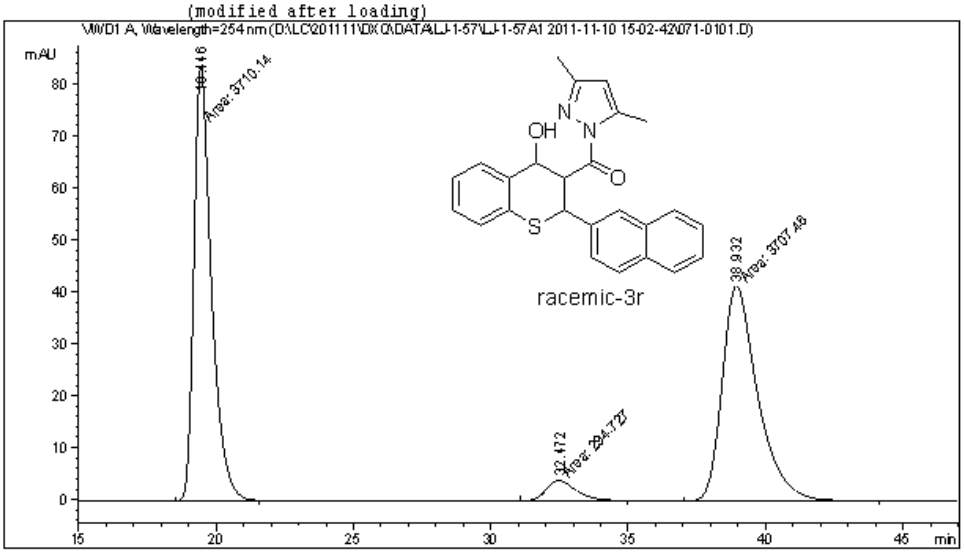



```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs

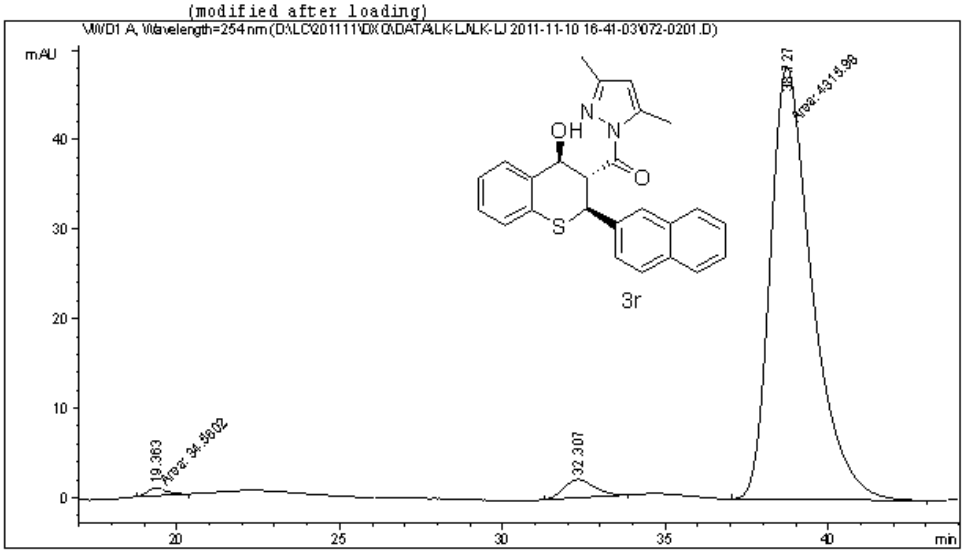

Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  19.446 MM    0.7419 3710.13672   83.34570  48.1066
   2  32.472 MM    1.2569  294.72723    3.90820   3.8215
   3  38.932 MM    1.4940 3707.45801   41.36030  48.0719

Totals :                  7712.32196  128.61420
```

Data File D:\LC\201111\DXQ\DATA\LK-LJ\LK-LJ 2011-11-10 16-41-03\072-0201.D
Sample Name: LJ-1-57B

Acq. Operator	: THL	Seq. Line	: 2
Acq. Instrument	: Instrument 1	Location	: Vial 72
Injection Date	: 11/10/2011 5:18:54 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201111\dxq\data\LK-LJ\LK-LJ 2011-11-10 16-41-03\ADH-10-90-1ML-254NM-45MIN.M		
Last changed	: 11/6/2011 7:43:25 PM by thl		
Analysis Method	: D:\LC\201111\DXQ\DATA\LK-LJ\LK-LJ 2011-11-10 16-41-03\072-0201.D\DA.M (ADH-10-90-1ML-254NM-45MIN.M)		
Last changed	: 11/10/2011 8:42:42 PM by THL (modified after loading)		



Area Percent Report

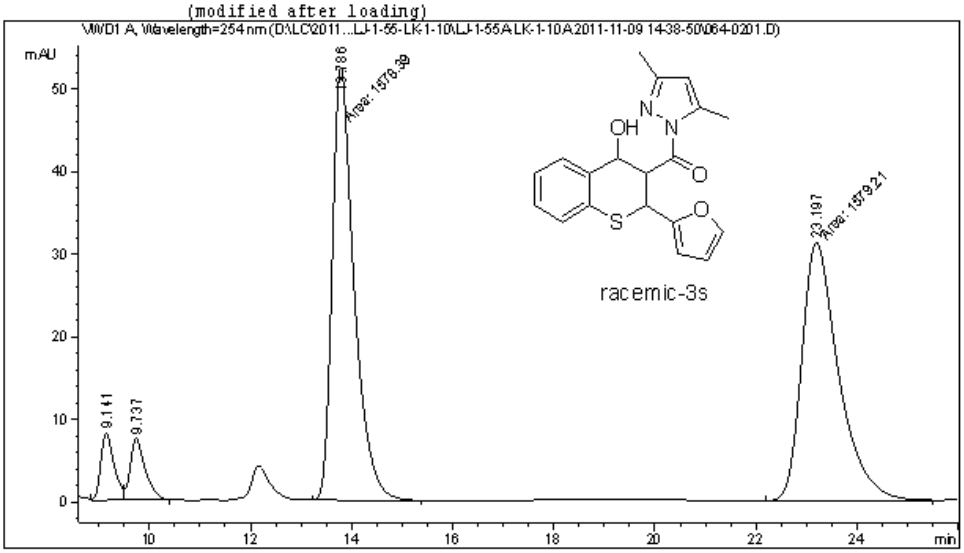
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	19.363	MM	0.6892	34.56019	8.35790e-1	0.7709
2	32.307	BB	0.8108	132.27974	2.03499	2.9508
3	38.727	MM	1.4880	4315.98340	48.34136	96.2782
Totals :				4482.82333	51.21214	

Data File D:\LC\2011...TA\LJ-1-55-LK-1-10\LJ-1-55A-LK-1-10A 2011-11-09 14-38-50\064-0201.D
Sample Name: LK-1-10A

Acq. Operator	: THL	Seq. Line	: 2
Acq. Instrument	: Instrument 1	Location	: Vial 64
Injection Date	: 11/9/2011 3:37:05 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\2011l1\dxq\data\LJ-1-55-LK-1-10\LJ-1-55A-LK-1-10A 2011-11-09 14-38-50\ADH-10-90-1ML-254NM-40MIN.M		
Last changed	: 11/9/2011 3:37:28 PM by THL (modified after loading)		
Analysis Method	: D:\LC\2011l1\DXQ\DATA\LJ-1-55-LK-1-10\LJ-1-55A-LK-1-10A 2011-11-09 14-38-50\064-0201.D\DA.M (ADH-10-90-1ML-254NM-40MIN.M)		
Last changed	: 11/10/2011 9:13:30 PM by THL (modified after loading)		



Area Percent Report

Sorted By	: Signal
Multiplier	: 1.0000
Dilution	: 1.0000

Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

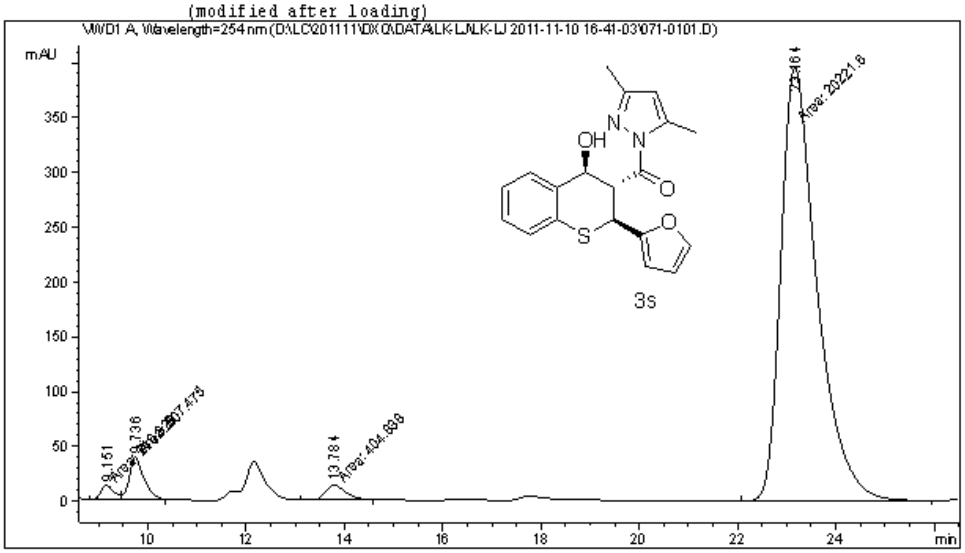
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	9.141	BV	0.2755	148.98314	8.10752	4.3061
2	9.737	VB	0.3038	153.23900	7.41240	4.4291
3	13.786	MM	0.5023	1578.39233	52.37198	45.6206
4	23.197	MM	0.8416	1579.21045	31.27554	45.6442
Totals :				3459.82492	99.16745	

Instrument 1 11/10/2011 9:13:34 PM THL

Page 1 of 2

Data File D:\LC\201111\DXQ\DATA\LK-LJ\LK-LJ 2011-11-10 16-41-03\071-0101.D
Sample Name: LK-1-10B

Acq. Operator	: THL	Seq. Line	: 1
Acq. Instrument	: Instrument 1	Location	: Vial 71
Injection Date	: 11/10/2011 4:42:15 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201111\dxq\data\LK-LJ\LK-LJ 2011-11-10 16-41-03\ADH-10-90-1ML-254NM-40MIN.M		
Last changed	: 11/10/2011 4:47:25 PM by THL (modified after loading)		
Analysis Method	: D:\LC\201111\DXQ\DATA\LK-LJ\LK-LJ 2011-11-10 16-41-03\071-0101.D\DA.M (ADH-10-90-1ML-254NM-40MIN.M)		
Last changed	: 11/10/2011 9:21:49 PM by THL (modified after loading)		



Area Percent Report

Sorted By	:	Signal	
Multiplier	:	1.0000	
Dilution	:	1.0000	

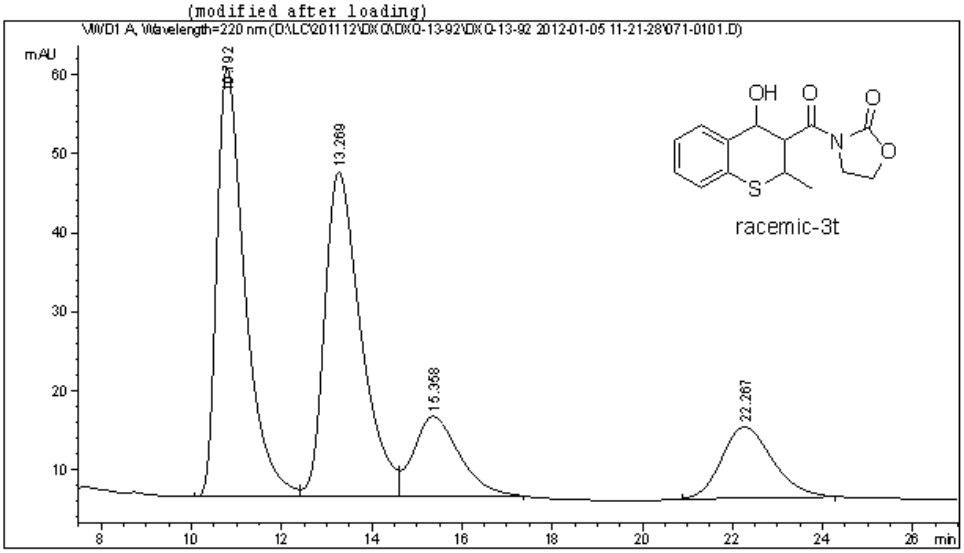
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU*s	Height [mAU]	Area %
1	9.151	MF	0.3056	240.32805	13.10678	1.1088
2	9.736	FM	0.3492	807.47461	38.53661	3.7255
3	13.784	MM	0.4819	404.83780	14.00192	1.8678
4	23.164	MM	0.8524	2.02216e4	395.39734	93.2978
Totals :				2.16742e4	461.04265	

Data File D:\LC\201112\DXQ\DXQ-13-92\DXQ-13-92 2012-01-05 11-21-28\071-0101.D
Sample Name: DXQ-13-92A

Acq. Operator	: LQH	Seq. Line	: 1
Acq. Instrument	: Instrument 1	Location	: Vial 71
Injection Date	: 1/5/2012 11:22:49 AM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201112\dxq\DXQ-13-92\DXQ-13-92 2012-01-05 11-21-28\ODH-30-70-1ML-220NM-60MIN.M		
Last changed	: 1/5/2012 12:09:42 PM by LQH (modified after loading)		
Analysis Method	: D:\LC\201112\DXQ\DXQ-13-92\DXQ-13-92 2012-01-05 11-21-28\071-0101.D\DA.M (ASH-30-70-1ML-220NM-60MIN.M)		
Last changed	: 1/6/2012 8:54:06 AM by THL (modified after loading)		



Area Percent Report

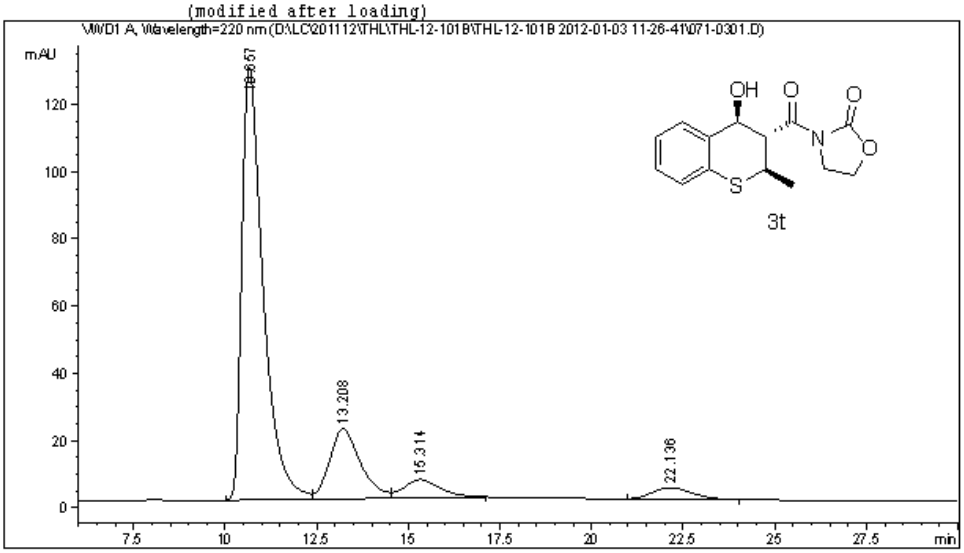
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	10.792	BB	0.6473	2351.31104	54.36254	38.6813
2	13.269	BV	0.8331	2294.43872	41.11519	37.7457
3	15.358	VB	1.0275	722.47992	10.17967	11.8855
4	22.267	BB	1.1195	710.44257	9.02372	11.6875
Totals :				6078.67224	114.68112	

Data File D:\LC\201112\THL\THL-12-101B\THL-12-101B 2012-01-03 11-26-41\071-0301.D
Sample Name: DXQ-13-91

Acq. Operator	: THL	Seq. Line	: 3
Acq. Instrument	: Instrument 1	Location	: Vial 71
Injection Date	: 1/3/2012 12:40:04 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201112\THL\THL-12-101B\THL-12-101B 2012-01-03 11-26-41\ODH-30-70-1ML-220-30MIN.M		
Last changed	: 1/3/2012 11:57:59 AM by LQH		
Analysis Method	: D:\LC\201112\THL\THL-12-101B\THL-12-101B 2012-01-03 11-26-41\071-0301.D\DA.M (ODH-30-70-1ML-220-30MIN.M)		
Last changed	: 1/6/2012 8:55:48 AM by THL (modified after loading)		



Area Percent Report

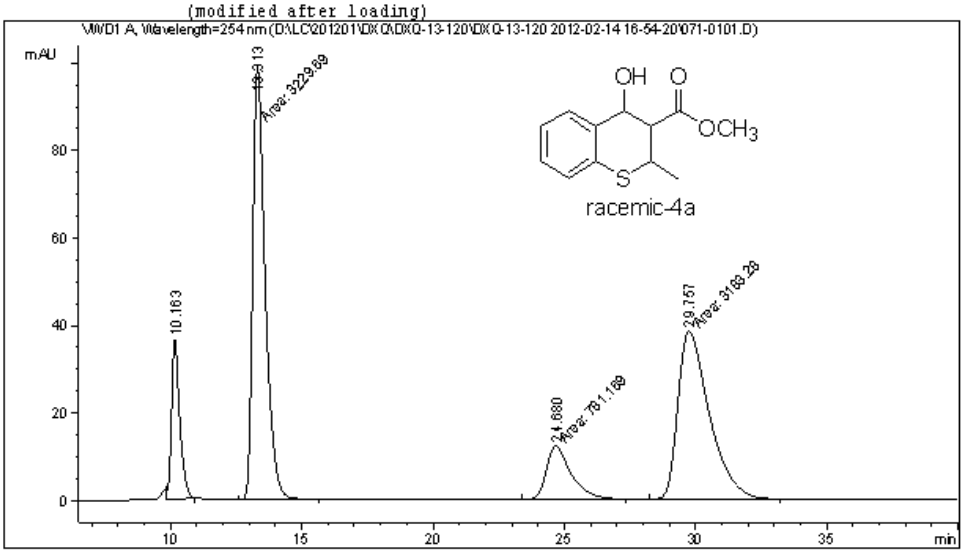
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	10.657	BB	0.6073	5208.14600	128.75288	73.4664
2	13.208	BV	0.8709	1230.11646	20.83359	17.3521
3	15.314	VB	0.9255	380.63272	5.44985	5.3692
4	22.136	BB	0.9277	270.25421	3.50313	3.8122
Totals :				7089.14938	158.53945	

Data File D:\LC\201201\DXQ\DXQ-13-120\DXQ-13-120 2012-02-14 16-54-20\071-0101.D
Sample Name: dxq-13-120

Acq. Operator	: thl	Seq. Line	: 1
Acq. Instrument	: Instrument 1	Location	: Vial 71
Injection Date	: 2/14/2012 4:55:31 PM	Inj	: 1
		Inj Volume	: 5 µl
Acq. Method	: D:\LC\201201\DXQ\DXQ-13-120\DXQ-13-120 2012-02-14 16-54-20\ASH-10-90-1ML-254NM.M		
Last changed	: 11/3/2011 11:58:38 AM by THL		
Analysis Method	: D:\LC\201201\DXQ\DXQ-13-120\DXQ-13-120 2012-02-14 16-54-20\071-0101.D\DA.M (ASH-10-90-1ML-254NM.M)		
Last changed	: 3/6/2012 8:09:48 PM by thl (modified after loading)		



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	10.163	VB	0.3223	790.82233	36.34251	9.9039
2	13.313	MM	0.5457	3229.68579	98.63870	40.4472
3	24.680	MM	1.0763	781.16901	12.09610	9.7830
4	29.757	MM	1.3841	3183.27539	38.33161	39.8659
Totals :				7984.95251	185.40893	

Data File D:\LC\201201\DXQ\DXQ-13-137\DXQ-13-137B 2012-03-02 11-06-58\085-0101.D
Sample Name: dxq-13-137b

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=====================================================================
Acq. Operator   : THL                          Seq. Line :   1
Acq. Instrument : Instrument 1                 Location : Vial 85
Injection Date  : 3/2/2012 11:18:33 AM              Inj :   1
Acq. Method     : D:\LC\201201\DXQ\DXQ-13-137\DXQ-13-137B 2012-03-02 11-06-58\ASH-10-90-1ML-
                  254NM-60MIN.M
Last changed    : 3/2/2012 11:34:22 AM by THL
                  (modified after loading)
Analysis Method : D:\LC\201201\DXQ\DXQ-13-137\DXQ-13-137B 2012-03-02 11-06-58\085-0101.D\DA.
                  M (ASH-10-90-1ML-254NM-60MIN.M)
Last changed    : 3/6/2012 8:15:35 PM by thl
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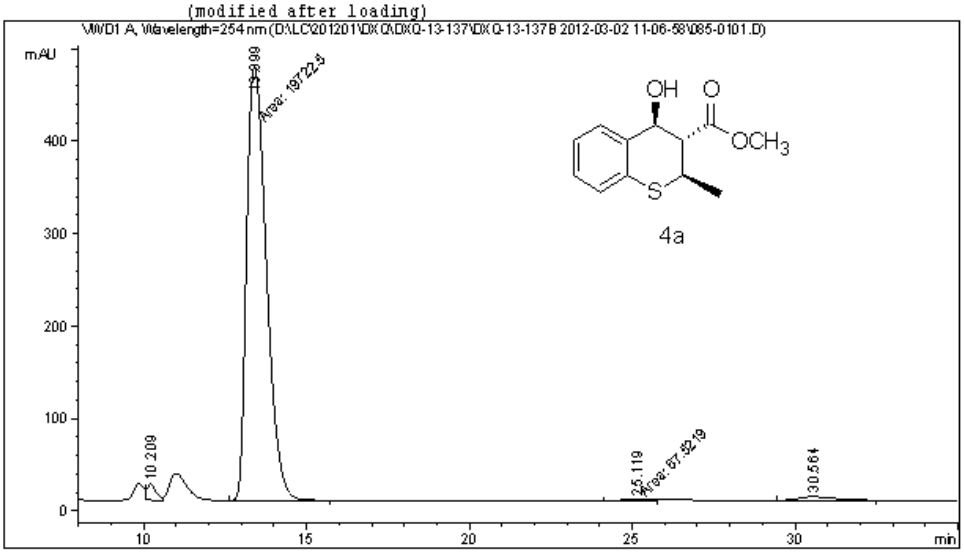


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=====================================================================
                        Area Percent Report
=====================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  10.209 VV    0.2794  336.21255   17.17018   1.6458
   2  13.399 MM    0.7016 1.97225e4   468.49265  96.5454
   3  25.119 MM    0.9260   67.52194    1.21532   0.3305
   4  30.564 BB    0.9068  301.98615    3.96818   1.4783

Totals :                  2.04282e4   490.84632
```