Microwave-Assisted Sequential One-Pot Protocol to Benzothiadiazin-3-one-1,1-dioxides via a Copper Catalyzed *N*-Arylation Strategy

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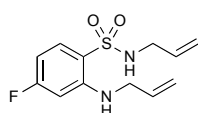
Table 2, Entry 8.

FTIR (neat): 3400, 1579, 1301, 1149, 547 cm^{-1}; ^{1}H NMR (400 MHz, CDCl$_3$) δ 7.73 (dd, *J* = 8.5, 6.6 Hz, 1H), 6.42 (ddd, *J* = 13.5, 9.7, 2.0 Hz, 2H), 6.19 (s, 1H), 5.98 – 5.83 (m, 1H), 5.67 (qt, *J* = 15.0, 7.5 Hz, 1H), 5.25 (dd, *J* = 19.5, 13.8 Hz, 2H), 5.12 (dd, *J* = 25.7, 13.7 Hz, 2H), 4.67 (t, *J* = 5.6 Hz, 1H), 3.88 – 3.74 (m, 2H), 3.52 (t, *J* = 5.8 Hz, 2H); ^{13}C NMR (126 MHz, CDCl$_3$) δ 167.8, 165.8, 147.9 (dd, J_{C-F} = 13.1 Hz), 133.2, 132.7, 132.7, 132.6, 117.8, 116.9, 103.5, 103.3, 99.6, 99.4, 46.1; HRMS calculated for C$_{12}$H$_{16}$FN$_2$O$_2$S (M+H)$^+$ 271.0917; found 271.0923.
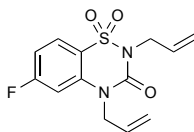
Scheme **4**, Entry **1** (R^1 = H, R^2 = Allyl, R^3 = Allyl).

FTIR (neat): 3400, 1575, 1310, 1149 cm^{-1}; ^{1}H NMR (500 MHz, CDCl$_3$) δ 7.92 – 7.83 (m, 1H), 7.03 – 6.94 (m, 2H), 6.04 – 5.88 (m, 2H), 5.39 – 5.30 (m, 2H), 5.29 – 5.19 (m, 2H), 4.69 – 4.59 (m, 2H), 4.52 – 4.46 (m, 2H); ^{13}C NMR (126 MHz, CDCl$_3$) δ 166.7, 164.6, 150.2, 138.8 (dd, J_{C-F} = 10.8 Hz), 131.6, 130.7, 125.3, 125.2, 119.2, 118.0, 111.1, 111.0, 104.7, 104.4, 48.5, 44.8; HRMS calculated for C$_{13}$H$_{14}$FN$_2$O$_3$S (M+H)$^+$ 297.0709; found 297.0712.
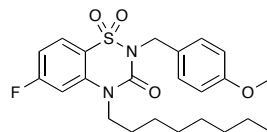
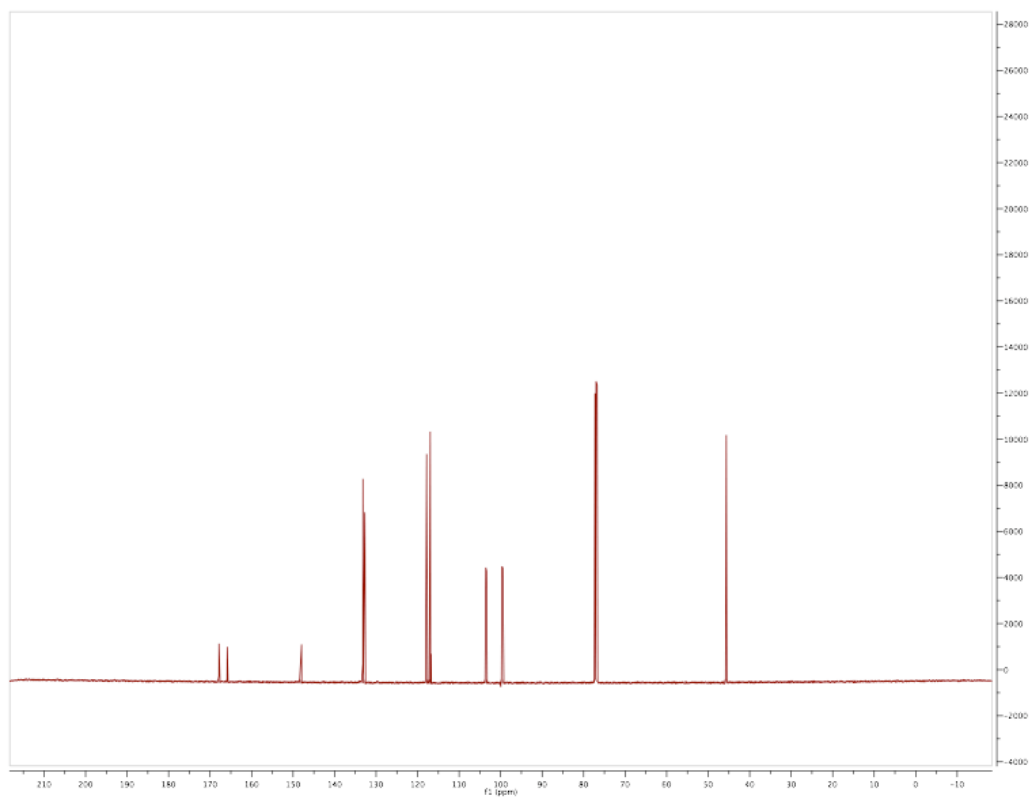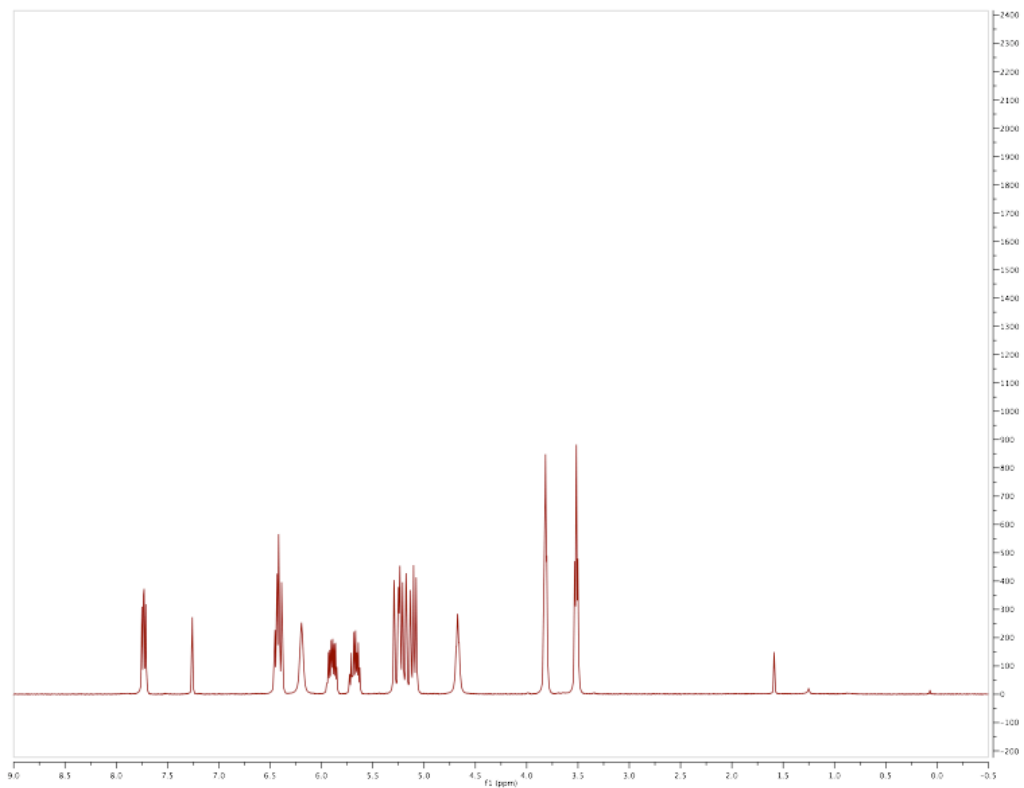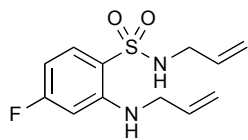Scheme **4**, Entry **2** (R^1 = F, R^2 = 4-MeOBn, R^3 = Octyl)

FTIR (neat) 3410, 1570, 1276, 1148 cm^{-1}: ^{1}H NMR (500 MHz, CDCl$_3$) δ = 7.91 (dd, *J* = 8.7, 5.8 Hz, 1H), 7.22 (dd, *J* = 10.2, 5.7 Hz, 1H), 7.02 – 6.96 (m, 3H), 6.94 (dd, *J* = 10.5, 2.2 Hz, 1H), 6.80 (ddd, *J* = 8.3, 2.6, 0.8 Hz, 1H), 5.03 (s, 2H), 4.01 – 3.97 (m, 2H), 3.76 (s, 3H), 1.71 (dt, *J* = 15.3, 7.6 Hz, 2H), 1.41 – 1.21 (m, 10H), 0.88 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (126 MHz, CDCl$_3$) δ = 165.9 (d, $^1J_{C-F}$ = 253.8 Hz), 159.63, 150.51, 138.6 (d, $^3J_{C-F}$ = 11.3 Hz),, 137.31, 129.55, 125.6 (d, $^3J_{C-F}$ = 9.6 Hz), 121.7, 120.89, 114.11, 113.53, 111.0 (d, $^2J_{C-F}$ = 24.2 Hz), 104.2 (d, $^2J_{C-F}$ = 29.0 Hz), 55.2, 45.9, 45.2, 31.7, 29.1, 29.1, 26.9, 26.5, 22.6, 14.1; HRMS calculated for C$_{23}$H$_{29}$FN$_2$O$_4$SNa (M + Na$^+$) 471.1730; found 471.1725 (TOF MS ES$^+$).
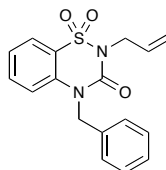
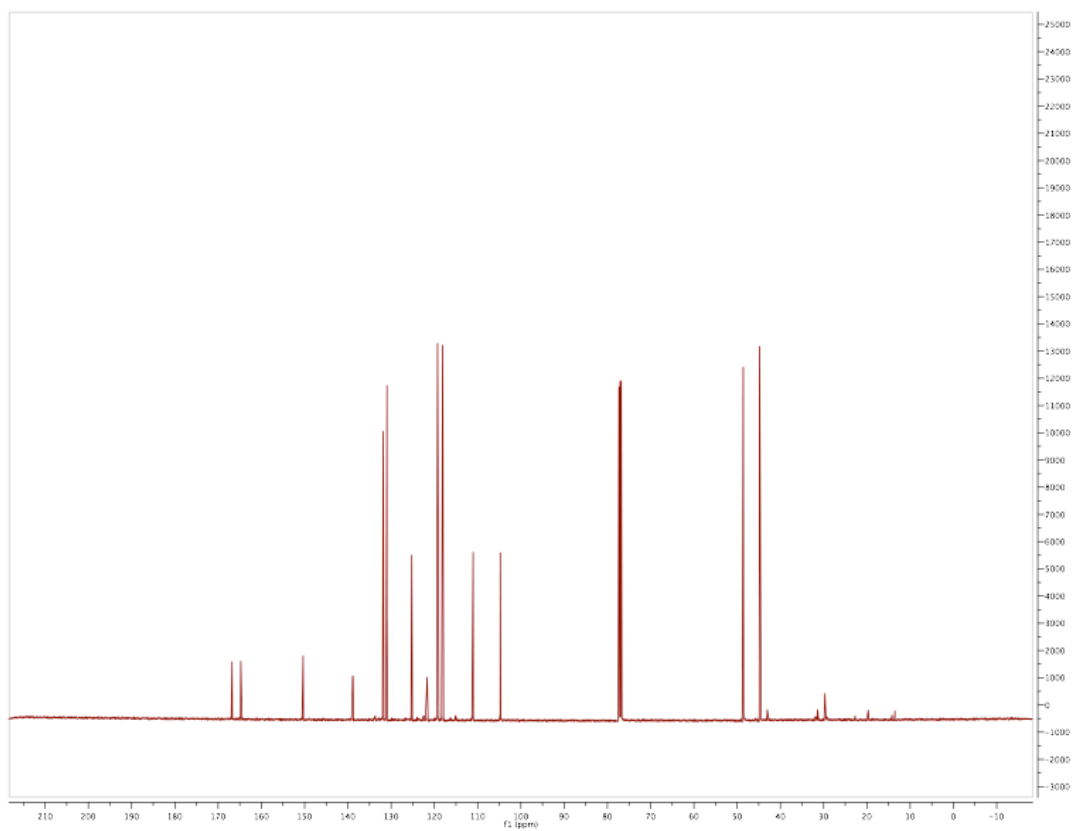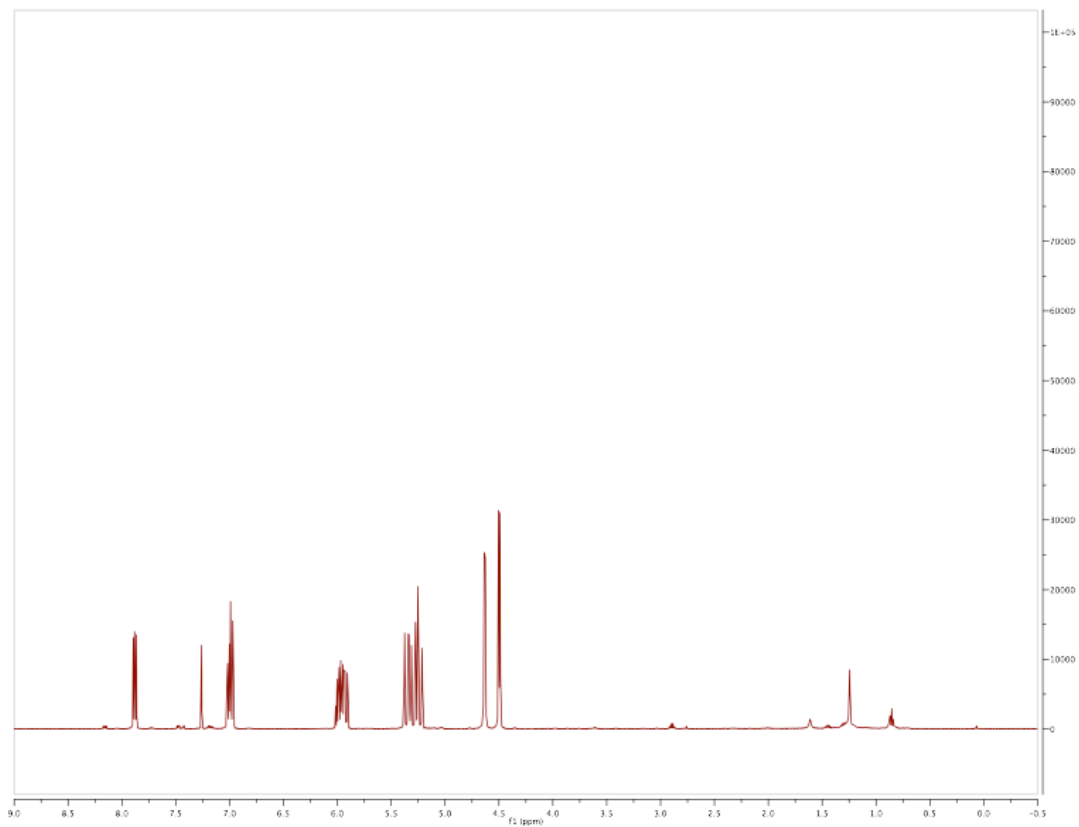Scheme **4**, Entry **3** (R^1 = F, R^2 = 4-MeOBn, R^3 = Octyl)

Using general procedure X, sultam **X** was produced in 89% (62 mg, 0.15 mmol) as a clear oil. FTIR (neat) 3401, 1571, 1315, 1148, 1163 cm^{-1}: ^{1}H NMR (400 MHz, CDCl$_3$) δ = 7.90 (dd, J = 7.8, 1.5 Hz, 1H), 7.54 – 7.48 (m, 1H), 7.38 – 7.32 (m, 2H), 7.31 – 7.23 (m, 4H), 7.14 (d, J = 8.4 Hz, 1H), 6.11 – 6.00 (m, 1H), 5.41 (ddd, J = 17.1, 2.6, 1.3 Hz, 1H), 5.34 (s, 2H), 5.33 – 5.28 (m, 1H), 4.59 (dt, J = 6.0, 1.2 Hz, 2H); ^{13}C NMR (126 MHz, CDCl$_3$) δ = 151.2, 136.4, 135.5, 134.1, 132.1, 129.0, 127.6, 126.2, 125.8, 123.7, 122.6, 119.1, 117.1, 49.1, 44.8; HRMS calculated for C$_{17}$H$_{16}$N$_2$O$_3$SNa (M + Na$^+$) 351.0779; found 351.0789 (TOF MS ES$^+$).
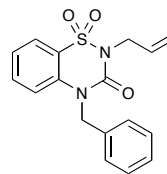
Table **2**, Entry **8**
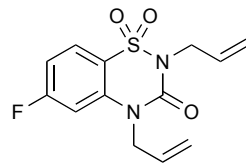
Scheme **4**, Entry **1** (R^1/R^2 = H, R^3 = Allyl, R^4 = Allyl).
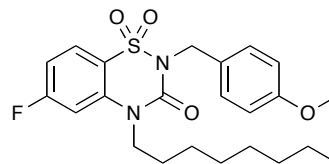
Scheme **4**, Entry **2** (R^1 = H, R^2 = F, R^3= 4-MeOBn, R^4 = Octyl).
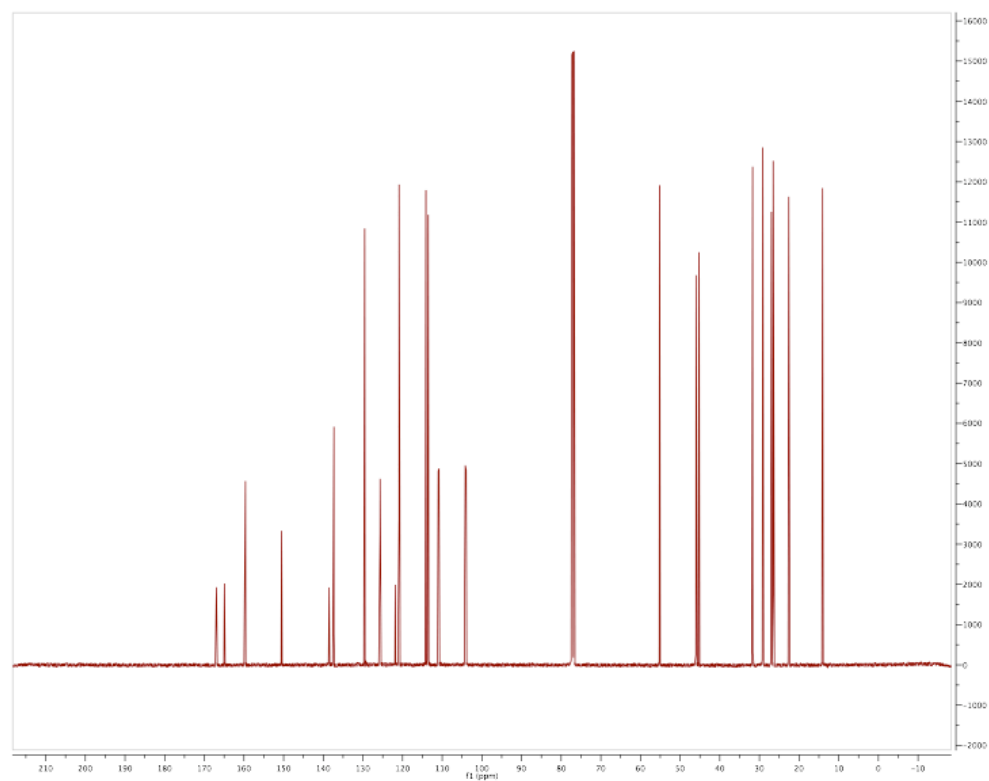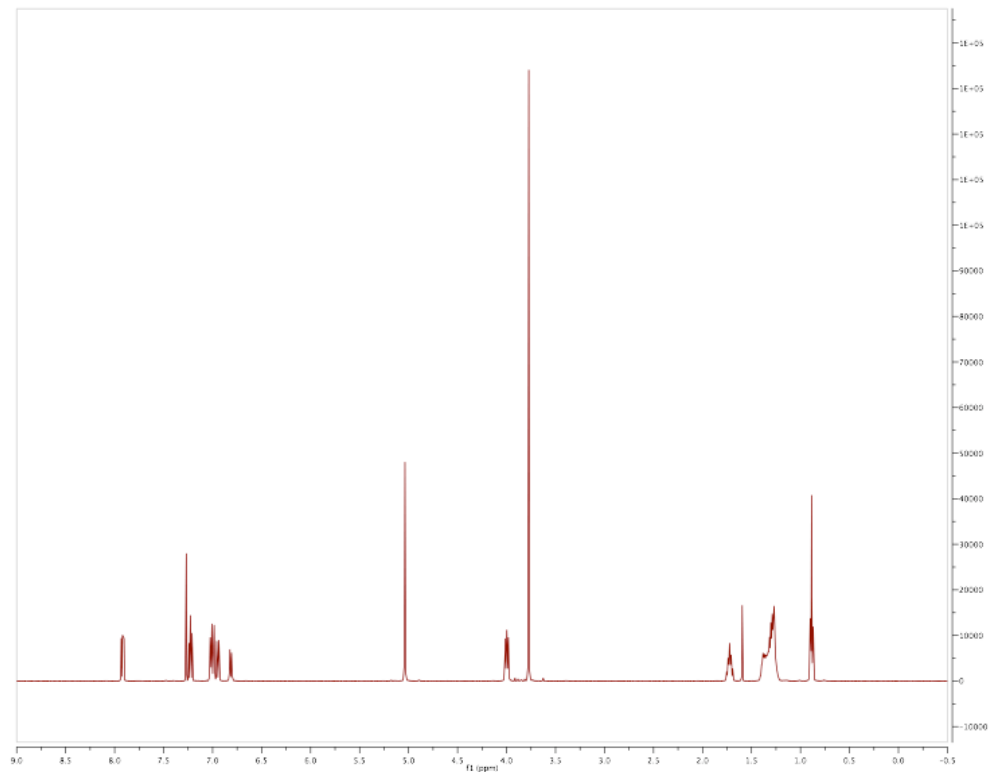
Scheme **4**, Entry **3** (R^1 = H, R^2 = F, R^3= 4-MeOBn, R^4 = Octyl)