



Supporting Information

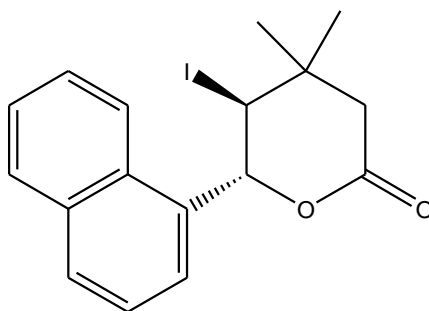
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Synthesis, Characterization, Cytotoxicity, and Antibacterial Properties of *trans*- γ -Halo- δ -lactones

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Agata Białońska,^[c] Weronika Gonciarz,^[d] and Magdalena Chmiela^[d]

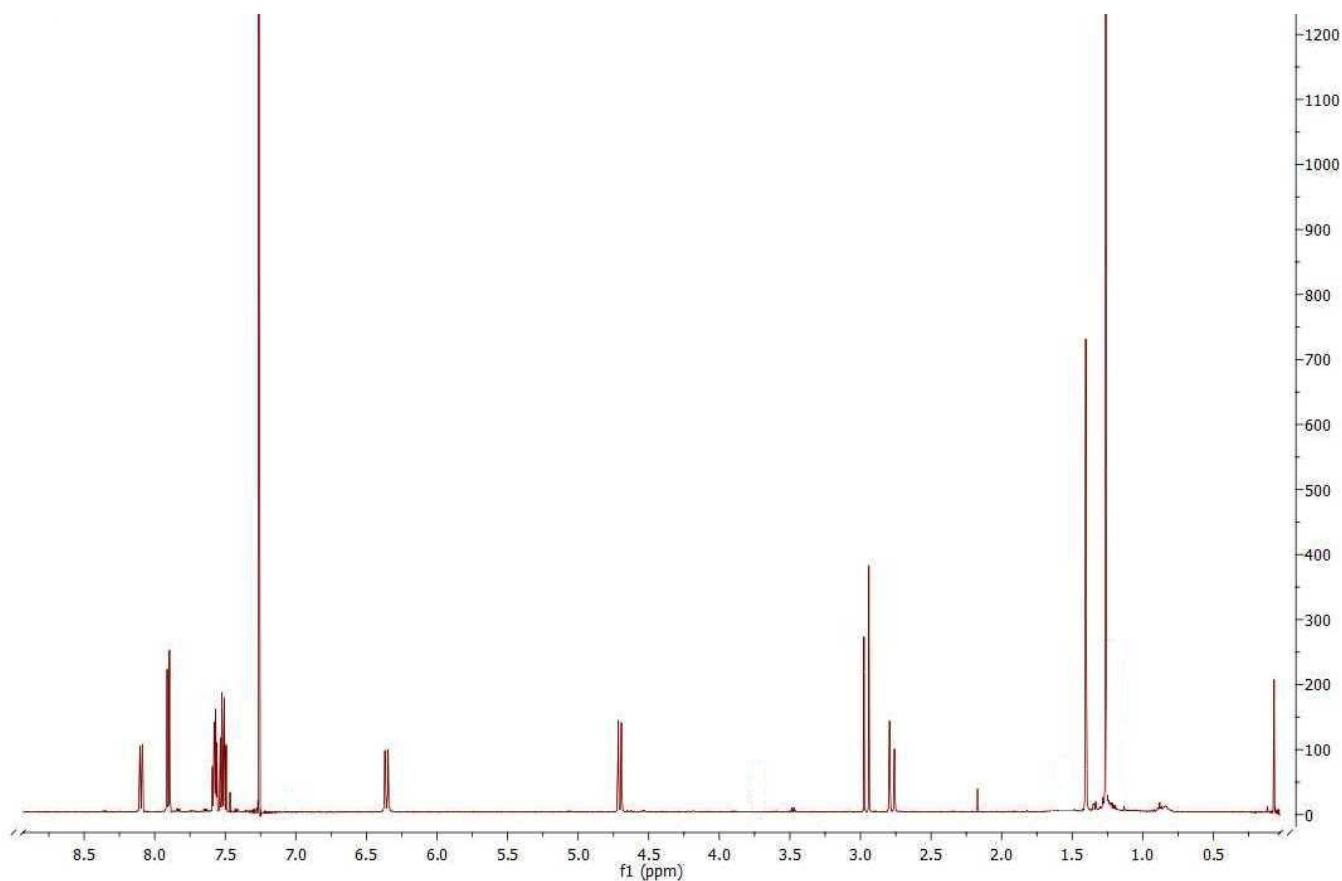
open_201800110_sm_miscellaneous_information.pdf

Spectral analysis of lactones **5-7a-c**

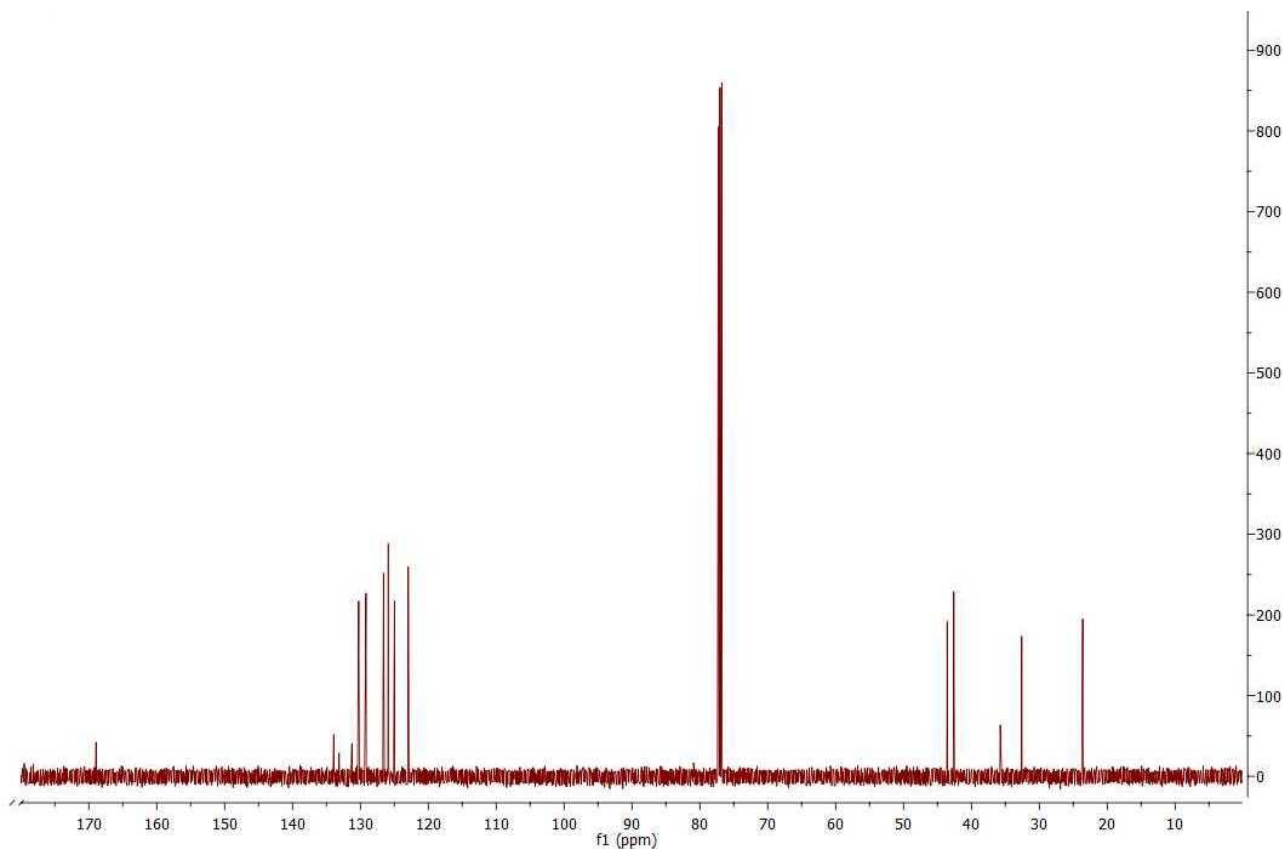


Compound 5a *trans*-5-iodo-4,4-dimethyl-6-(α -naphthyl) tetrahydro-2H-pyran-2-one

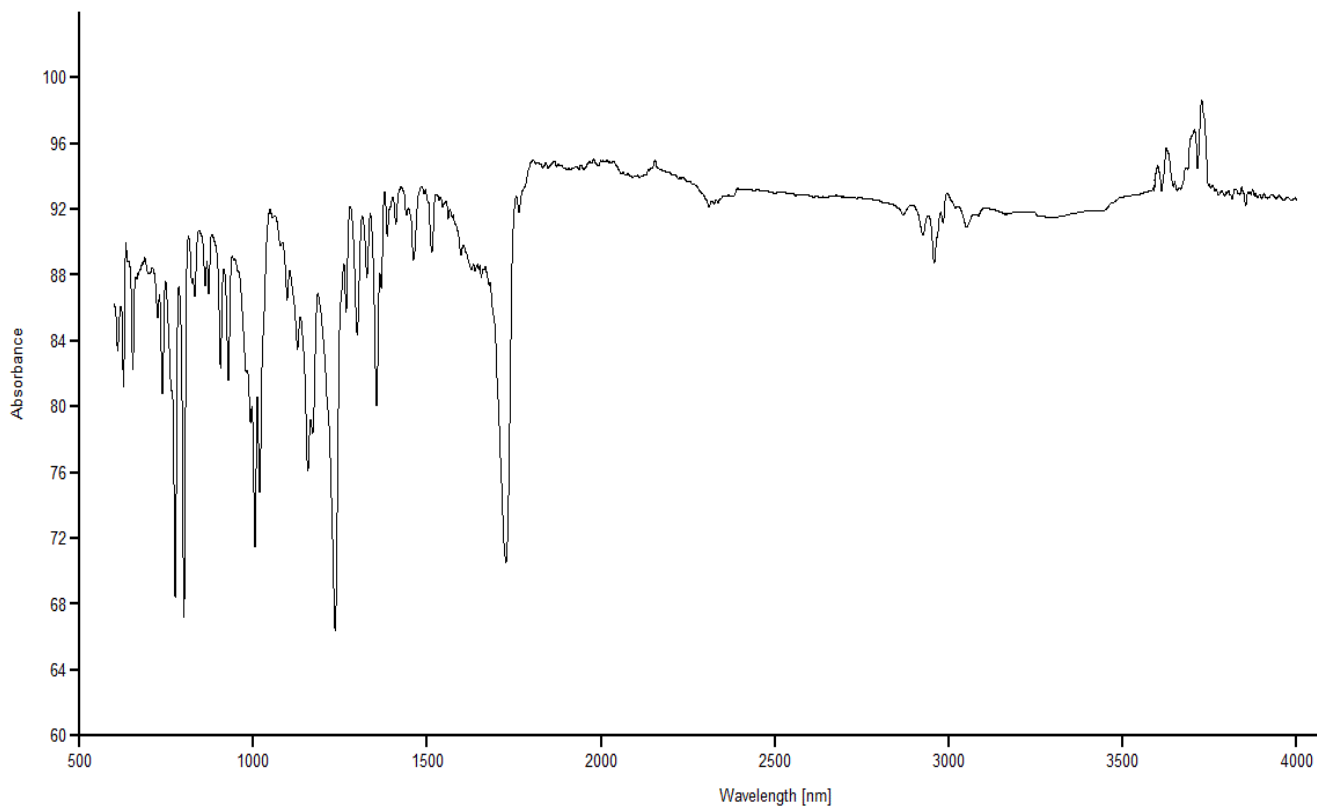
Yield 93%, yellow crystals, m.p. =118–119°C



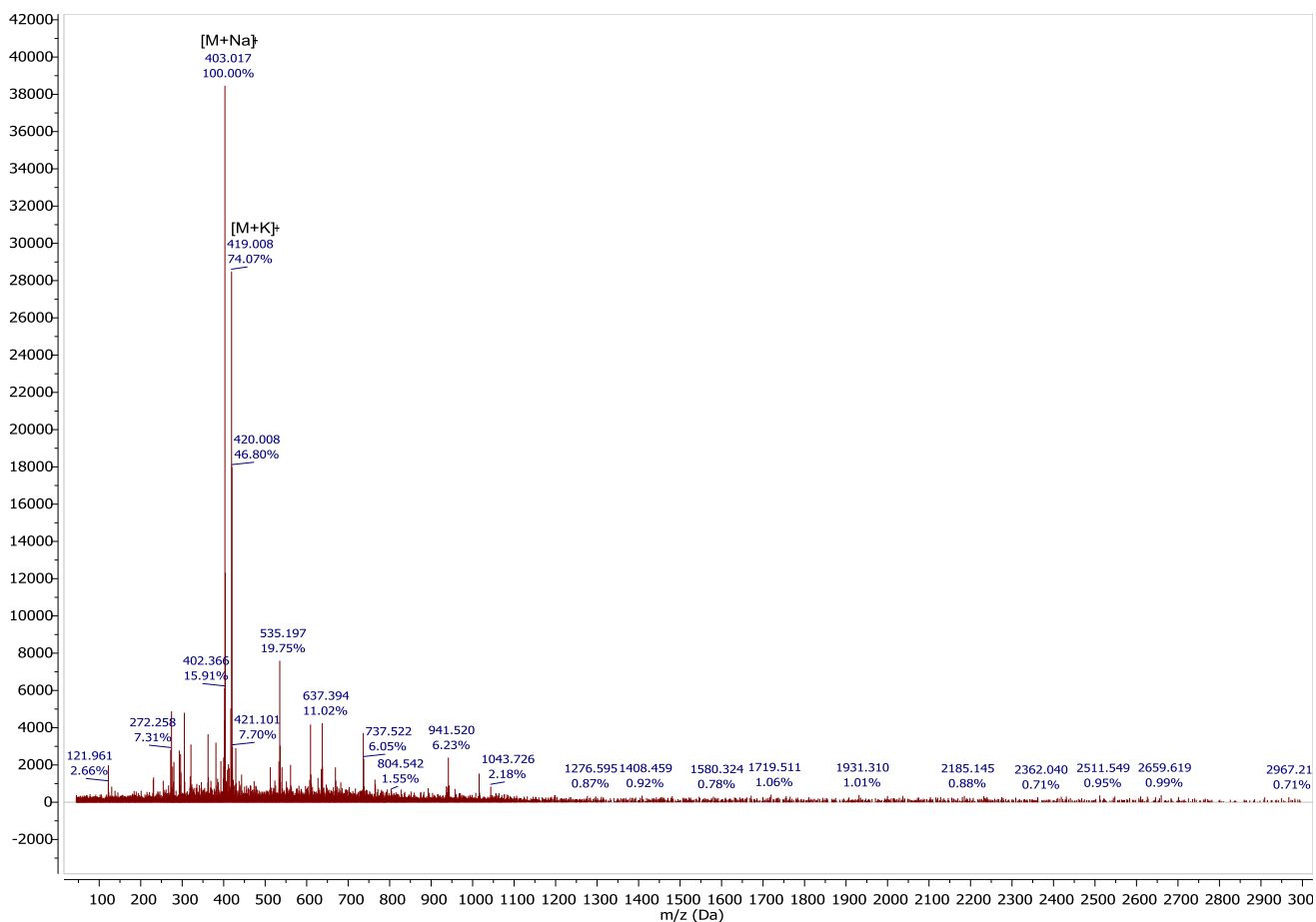
^1H NMR (CDCl_3 , 500 MHz) δ [ppm]: 1.26 (s, $\text{C}(\text{CH}_3)_2$, 3H), 1.40 (s, $\text{C}(\text{CH}_3)_2$, 3H), 2.78 (d, $J = 17.3$ Hz, CH_2 , 1H), 2.96 (d, $J = 17.3$ Hz, CH_2 , 1H), 4.71 (d, $J = 11.4$ Hz, CHI , 1H), 6.36 (d, $J = 11.4$ Hz, CHAr , 1H), 7.48–7.55 (m, HAr , 2H), 7.56–7.61 (m, HAr , 2H), 7.90 (d, $J = 8.2$ Hz, HAr , 2H), 8.10 (d, $J = 8.6$ Hz, HAr , 1H).



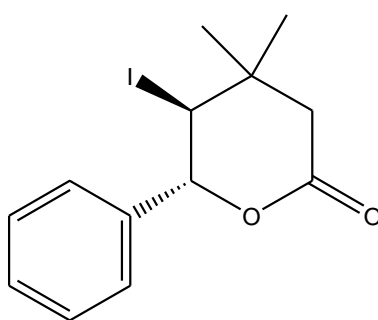
^{13}C NMR (125 Hz, CDCl_3) δ [ppm]: 23.60, 32.58, 35.75, 42.59, 43.57, 77.23, 122.95, 124.98, 125.90, 125.95, 126.60, 129.16, 130.4, 131.24, 133.13, 133.92, 168.94.



IR: 1723, 1352, 1235, 1155, 1020, 1002, 800, 775, 735, 650, 620, 610 cm^{-1} .

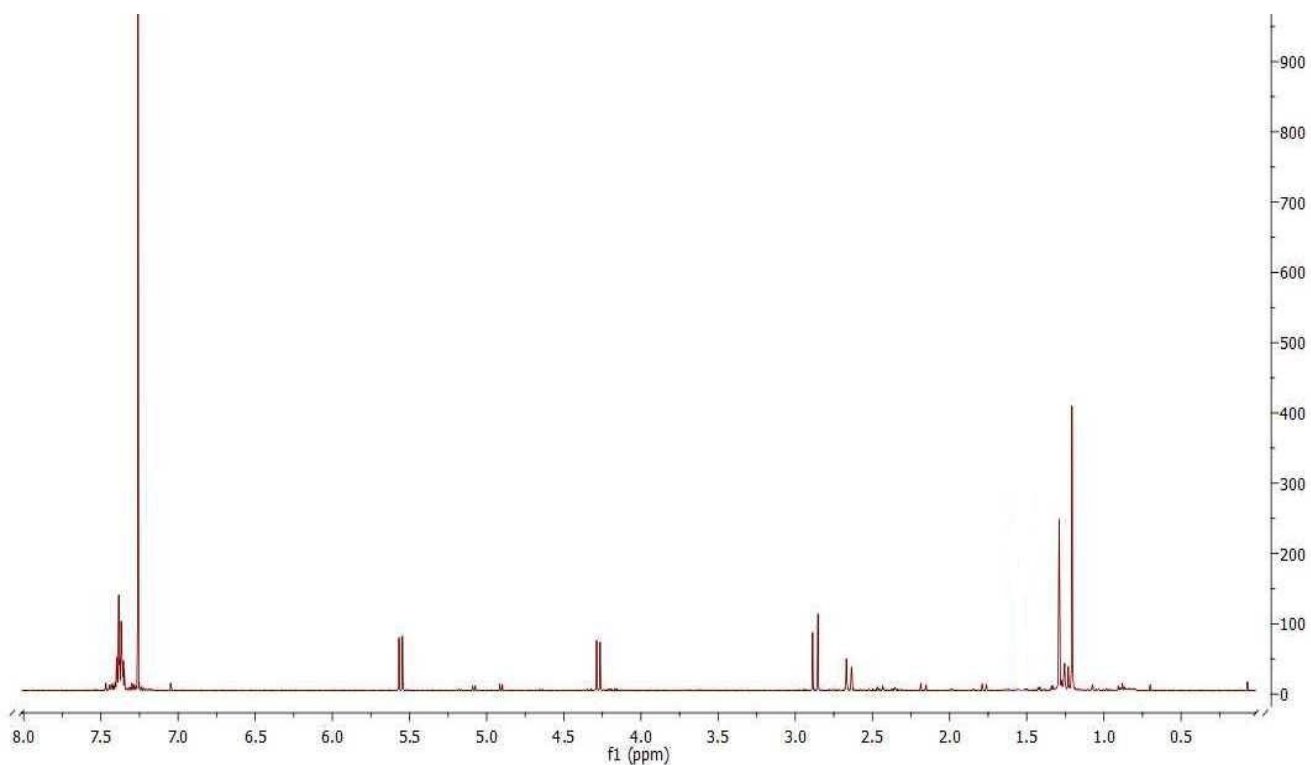


HR-MS (ESI-TOF) calculated for $C_{17}H_{17}IO_2$, m/z $[M+Na]^+$: 403.0170952; experimental value: 403.017215

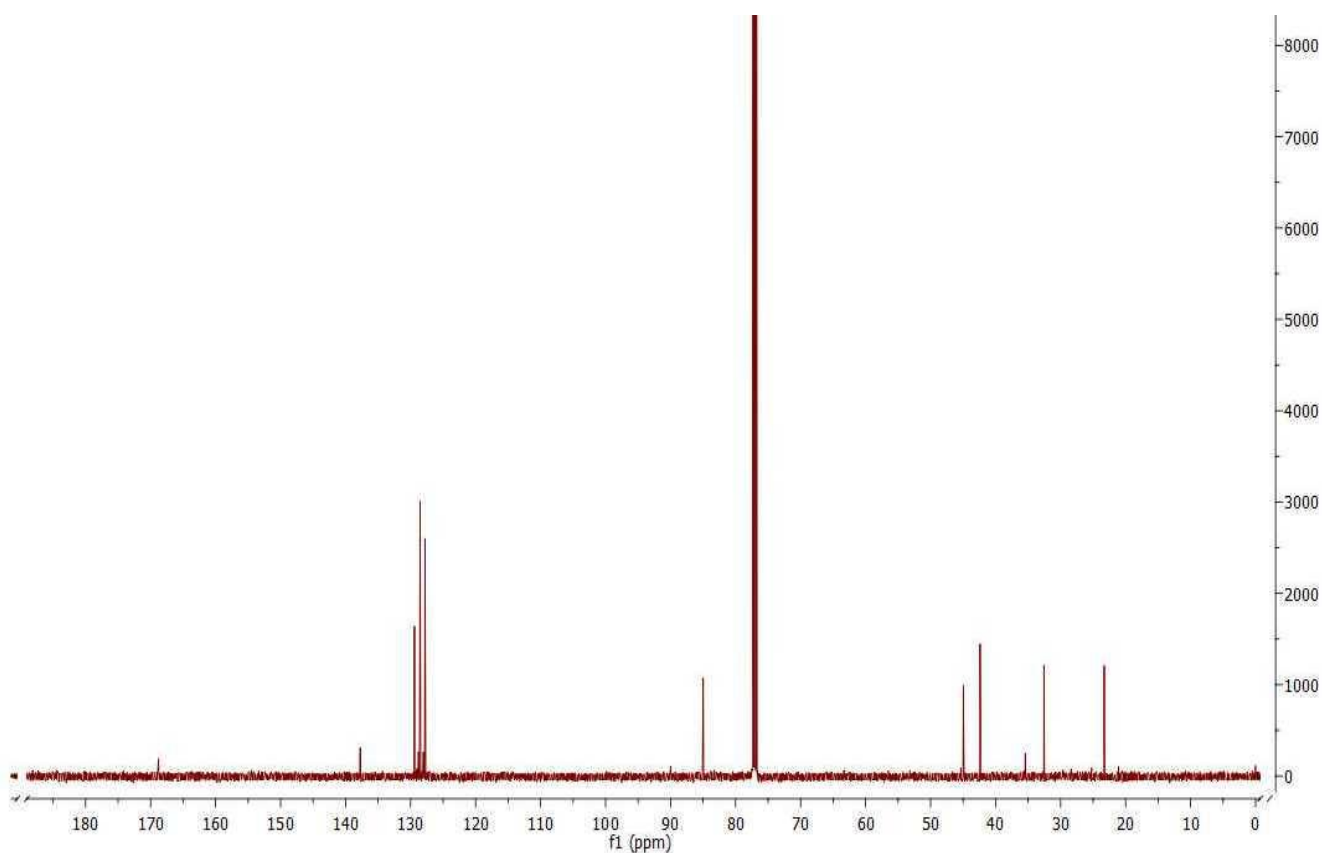


Compound 5b *trans*-5-iodo-4,4-dimethyl-6-phenyl-tetrahydro-2H-pyran-2-one

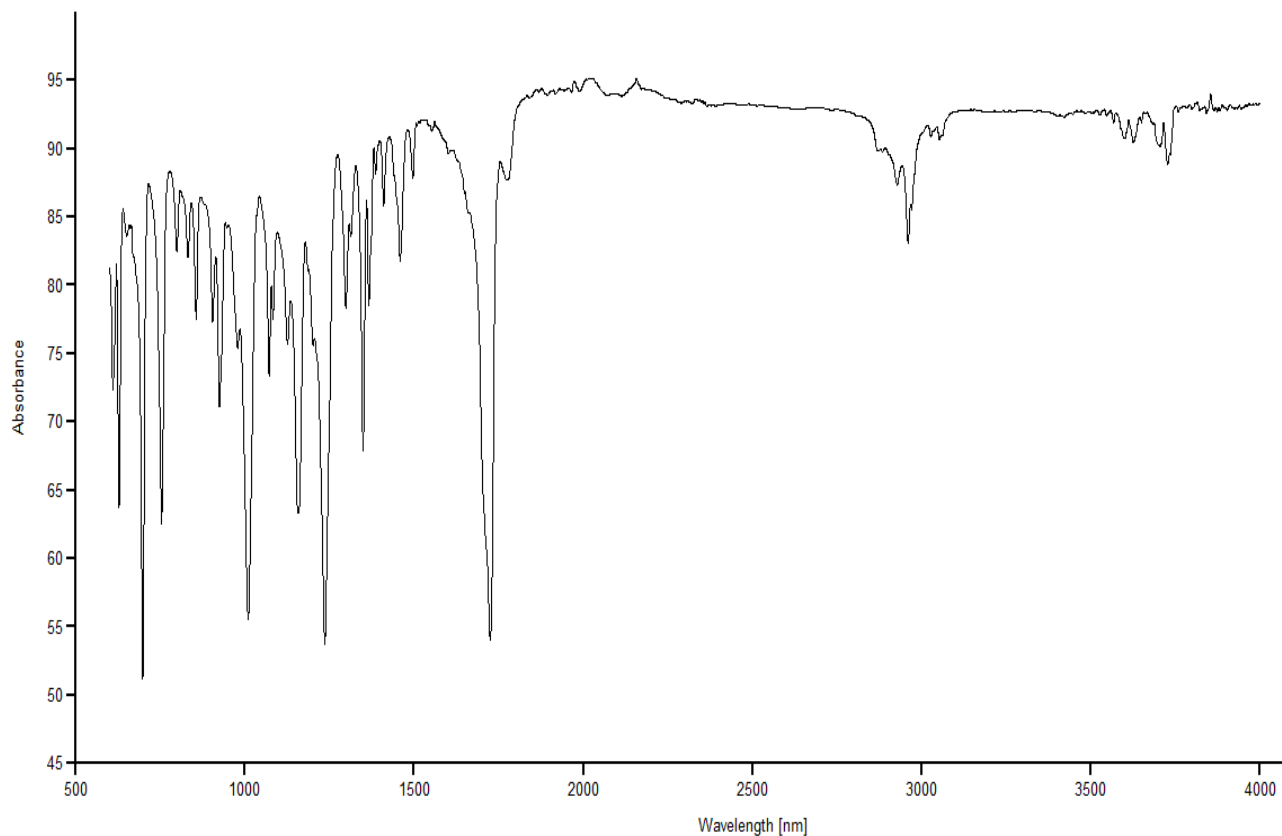
Yield 87%, yellow crystals, m.p. = 72–73°C,



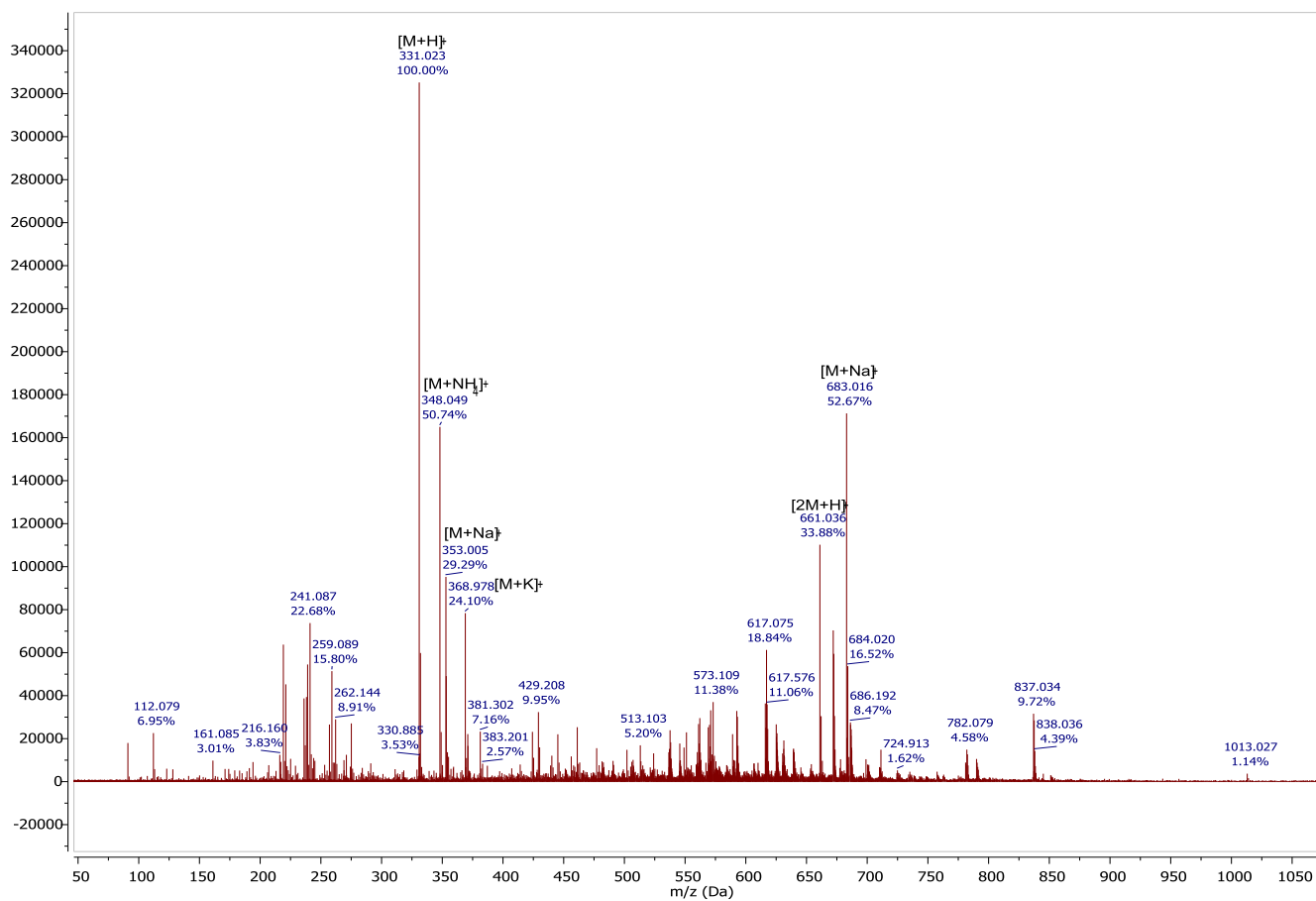
^1H NMR (CDCl_3 , 500 MHz), δ [ppm]: 1.21 (s, $\text{C}(\text{CH}_3)_2$, 3H), 1.29 (s, $\text{C}(\text{CH}_3)_2$, 3H), 2.65 (d, $J = 17.3$ Hz, CH_2 , 1H), 2.87 (d, $J = 17.3$ Hz, CH_2 , 1H), 4.28 (d, $J = 11.3$ Hz, CHBr , 1H), 5.56 (d, $J = 11.3$ Hz, CHAr , 1H), 7.34–7.48 (m, HAr , 5H).



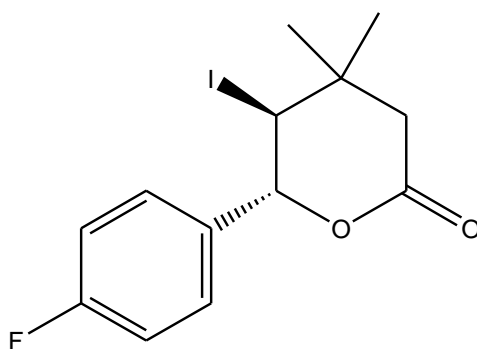
^{13}C NMR (125Hz, CDCl_3) δ [ppm]: 23.28, 32.57, 35.44, 42.38, 44.97, 84.98, 127.78, 128.53, 129.40, 137.75, 168.91.



IR: 1723, 1460, 1350, 1240, 1160, 1070, 1005, 925, 851, 750, 700, 620, 609 cm^{-1} .

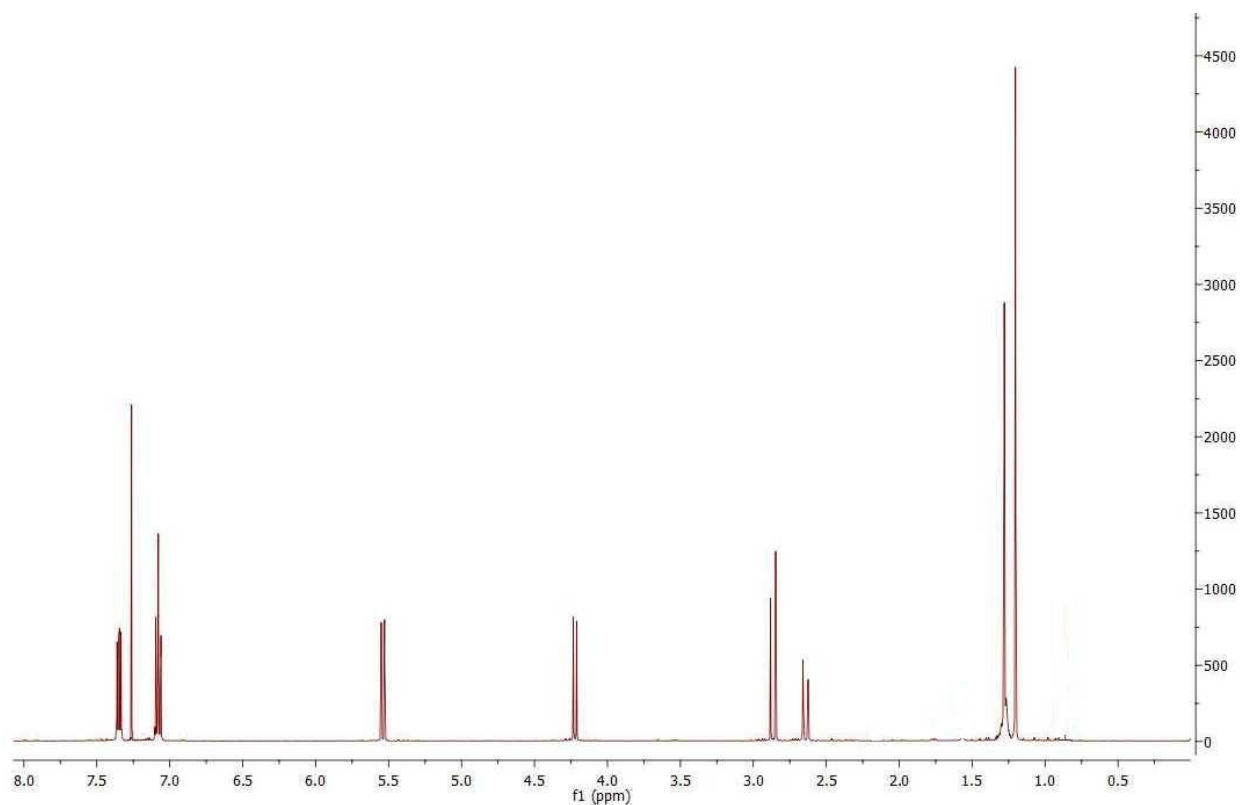


HR-MS (ESI-TOF) calculated for $\text{C}_{13}\text{H}_{15}\text{O}_2$, m/z [M+Na]⁺: 353.003446; experimental value: 353.004945.

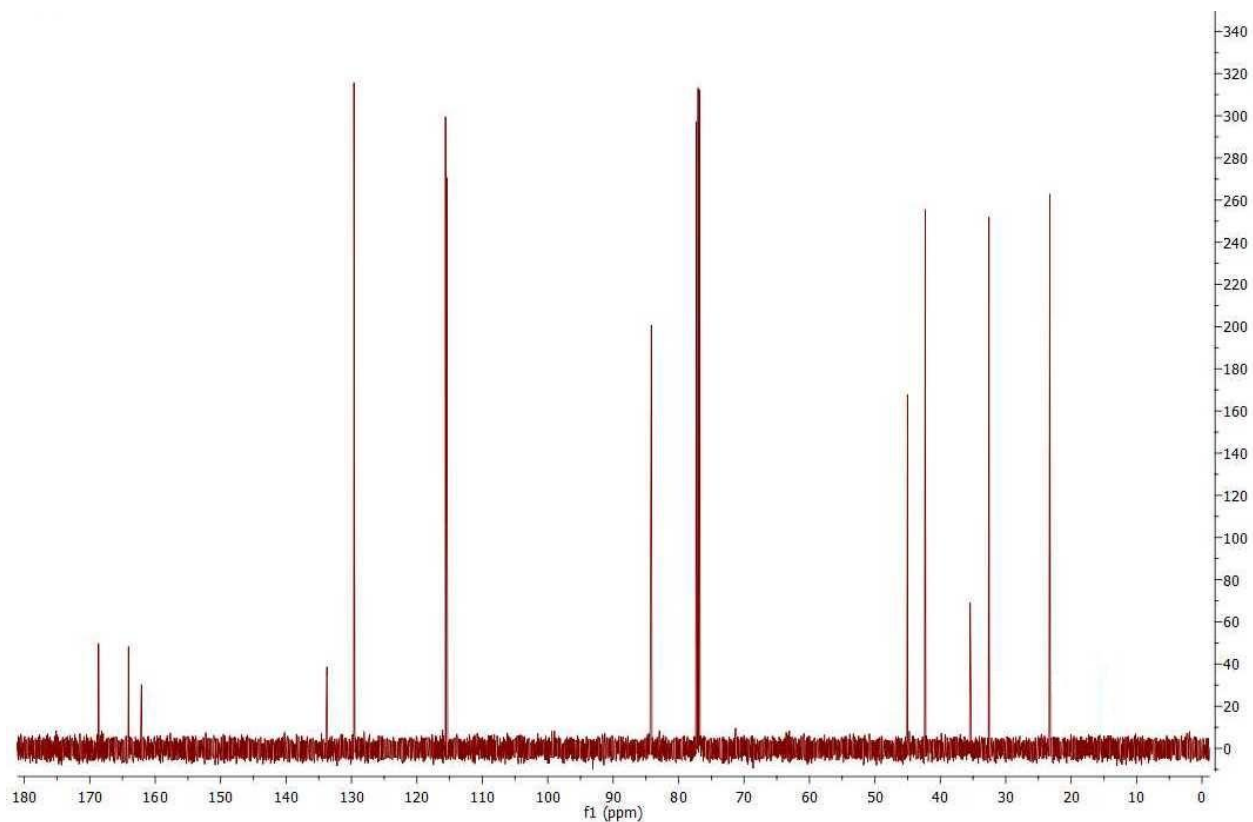


Compound 5c *trans*-6-(*p*-fluorophenyl)-5-iodo-4,4-dimethyltetrahydro-2H-pyran-2-one

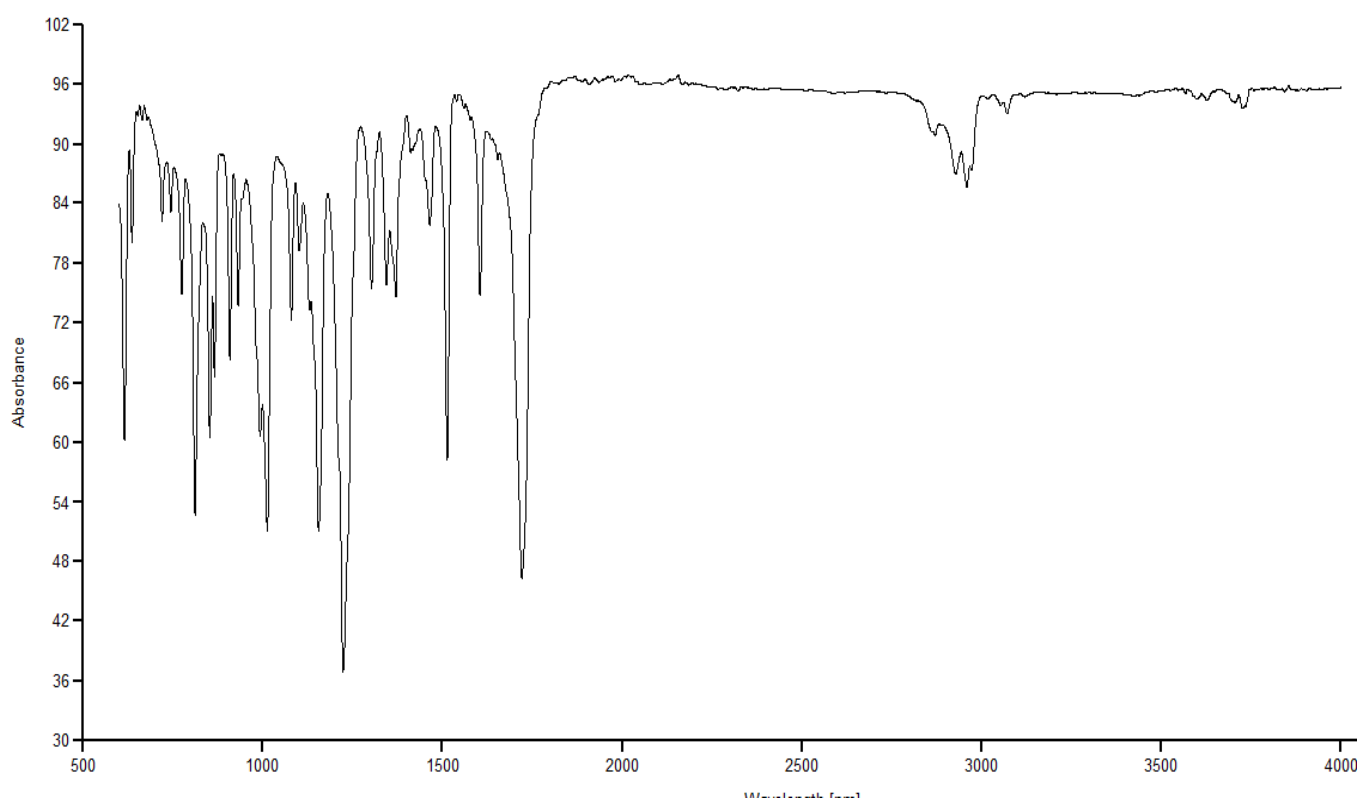
Yield 60%, yellow crystals, m.p. = 81–82°C,



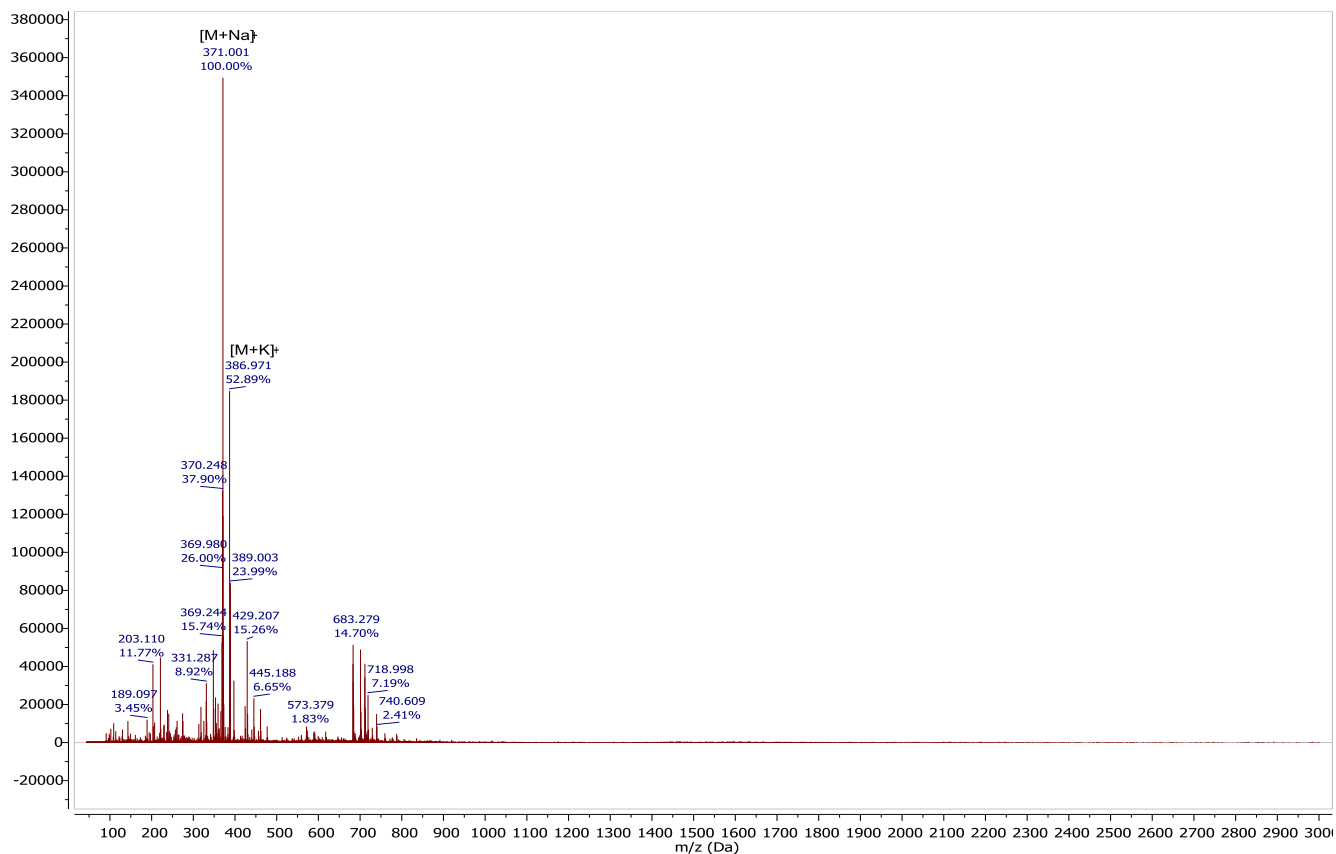
^1H NMR (CDCl_3 , 500 MHz) δ [ppm]: 1.20 (s, $\text{C}(\text{CH}_3)_2$, 3H), 1.28 (s, $\text{C}(\text{CH}_3)_2$, 3H), 2.64 (d, $J = 17.3$ Hz, CH_2 , 1H), 2.76 (d, $J = 17.3$ Hz, CH_2 , 1H), 4.22 (d, $J = 11.4$ Hz, CHI , 1H), 5.54 (d, $J = 11.4$ Hz, CHAr , 1H), 7.04–7.14 (m, HAr , 2H), 7.32–7.35 (m, HAr , 2H).



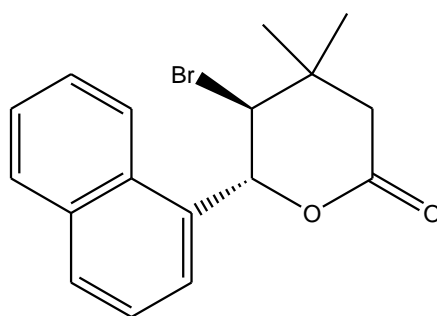
^{13}C NMR (125 Hz, CDCl_3) δ [ppm]: 23.26, 32.56, 35.43, 42.31, 45.01, 84.16, 115.54, 129.59, 133.77, 163.09, 168.65.



IR: 1720, 1605, 1515, 1368, 1342, 1300, 1220, 1154, 1078, 1009, 904, 850, 805, 615 cm^{-1} .

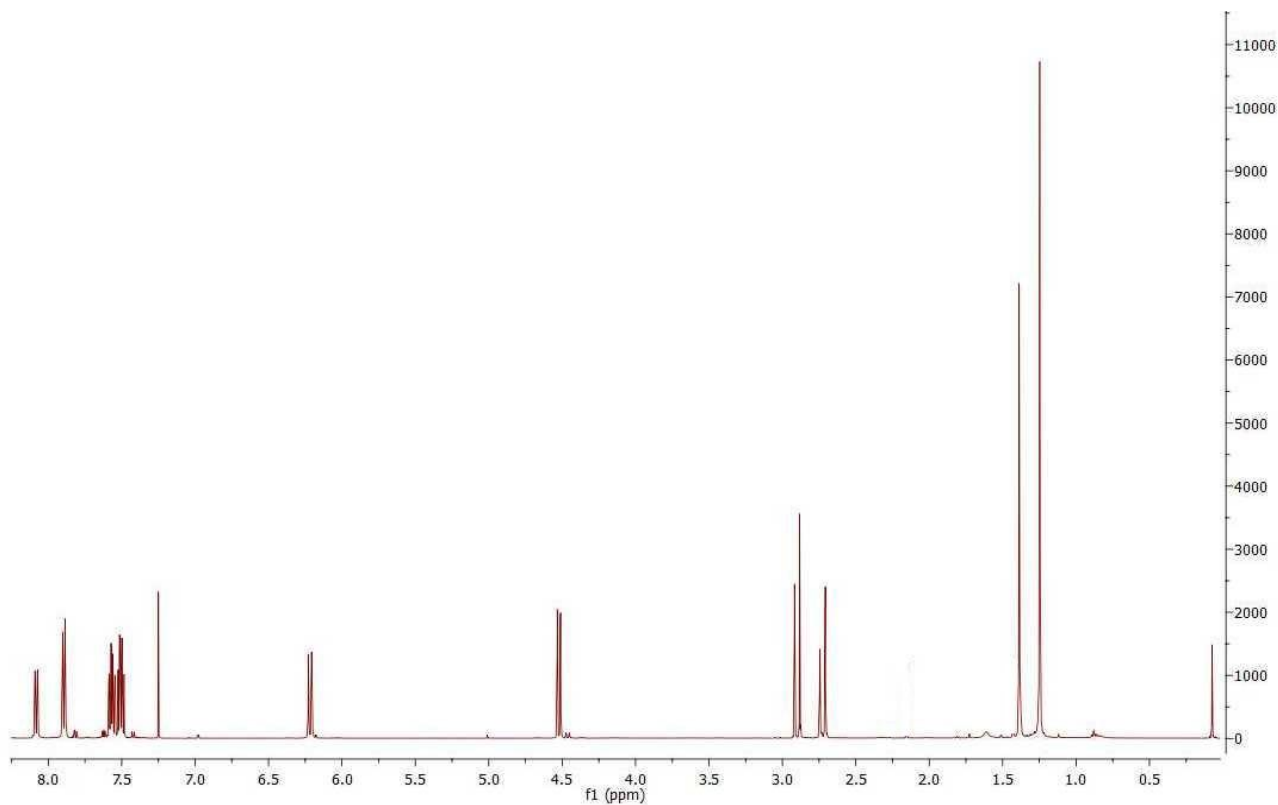


HR-MS (ESI-TOF) calculated for $C_{13}H_{14}FO_2$, m/z $[M+Na]^+$: 370.9920246; experimental value: 371.001004.

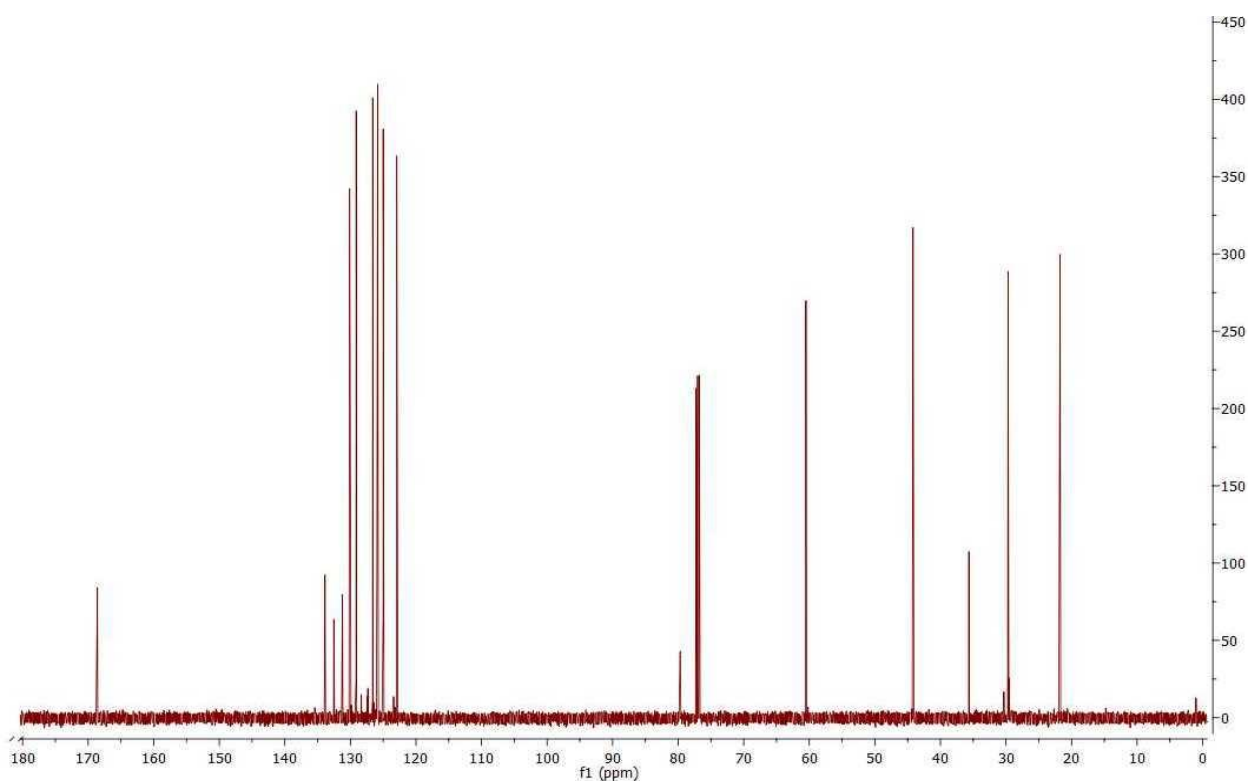


Compound 6a *trans*-5-bromo-4,4-dimethyl-6-(α -naphthyl) tetrahydro-2H-pyran-2-one

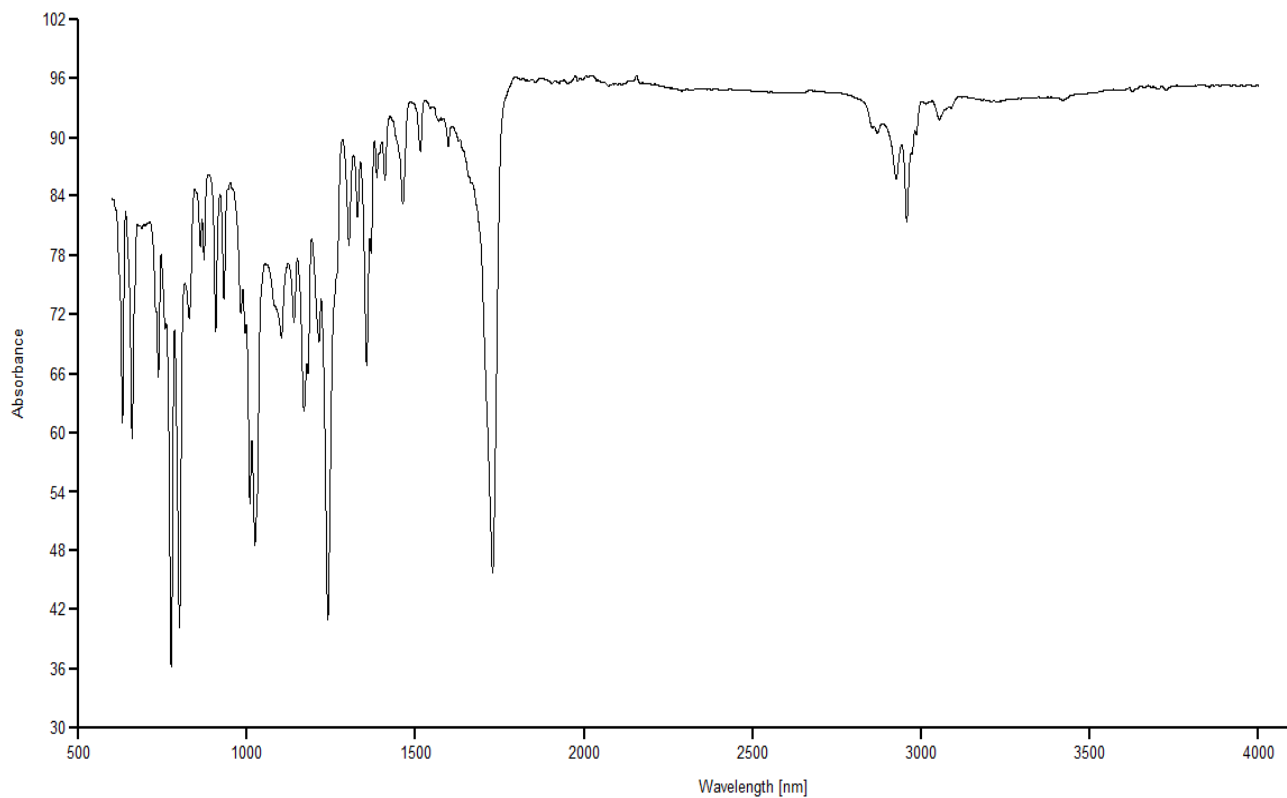
Yield 62%, colorless crystals, m.p. = 197–198°C,



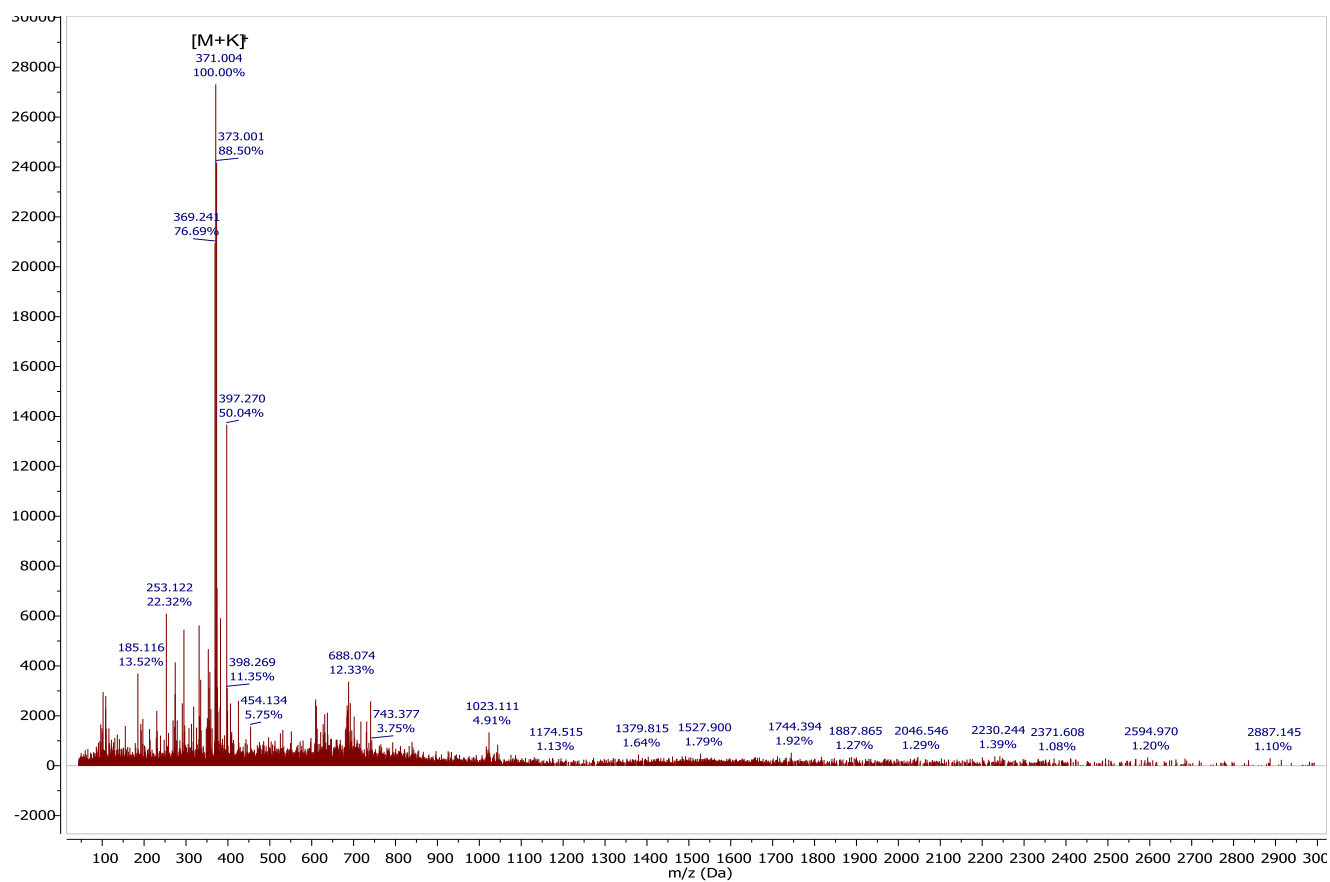
¹H NMR (CDCl₃, 500 MHz), δ [ppm]: 1.29 (s, C(CH₃)₂, 3H), 1.42 (s, C(CH₃)₂, 3H), 2.76 (d, *J* = 17.4 Hz, CH₂, 1H), 2.94 (d, *J* = 17.4 Hz, CH₂, 1H) 4.56 (d, *J* = 10.7 Hz, CHBr, 1H), 6.25 (d, *J* = 10.7 Hz, CHAr, 1H), 7.47–7.53 (m, HAr, 2H), 7.53–7.59 (m, HAr, 2H), 7.89 (d, *J* = 8.2 Hz, HAr, 2H), 8.08 (d, *J* = 8.6 Hz, HAr, 1H).



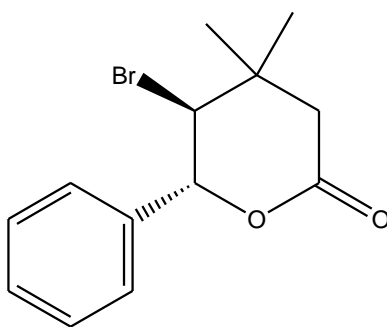
¹³C NMR (125 Hz, CDCl₃) δ [ppm]: 21.74, 29.68, 35.65, 44.18, 60.51, 79.69, 122.93, 125.00, 125.85, 125.95, 126.60, 129.12, 130.12, 131.22, 132.53, 133.88, 168.59



IR: 1730, 1460, 1355, 1240, 1160, 1100, 1020, 1007, 908, 802, 780, 740, 660, 630 cm^{-1} .

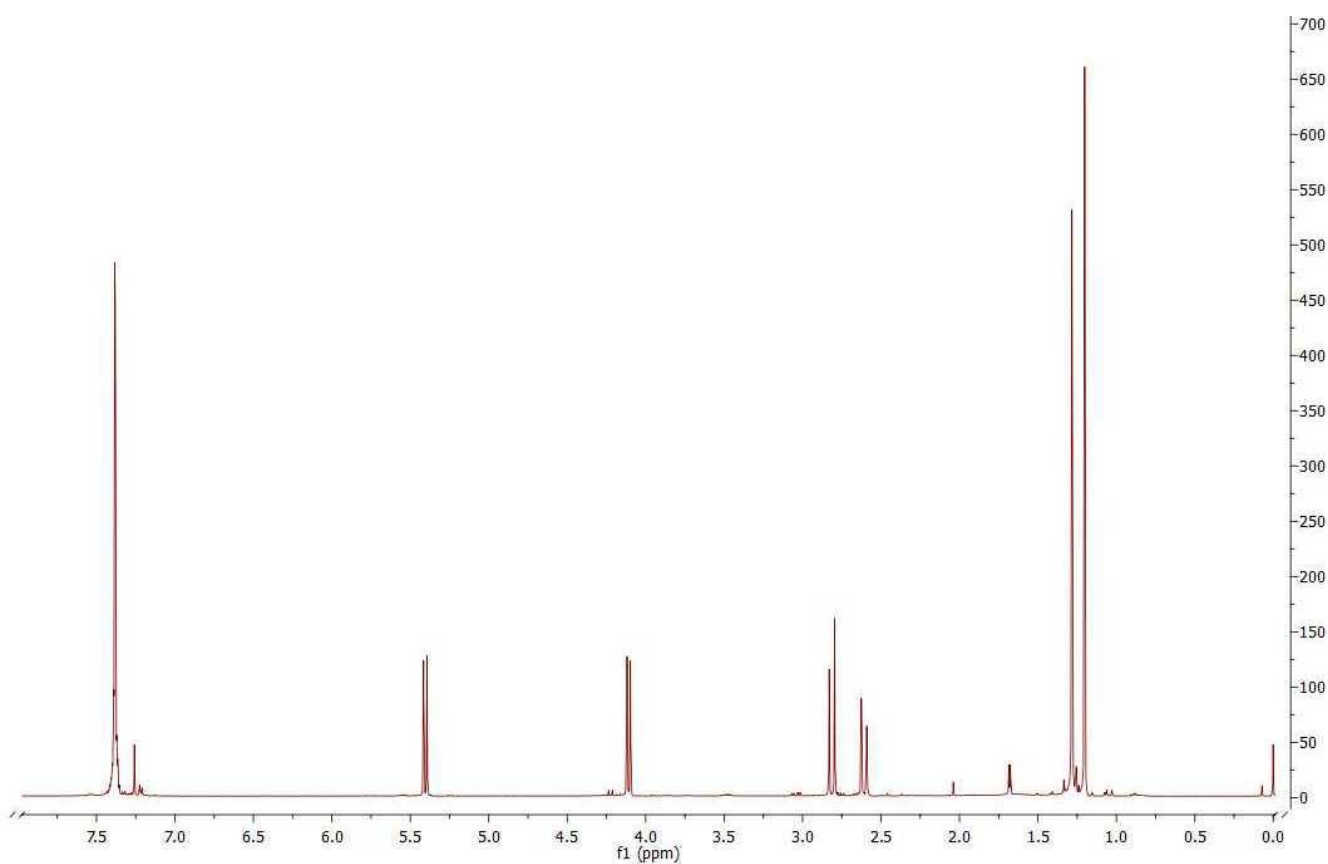


HR-MS (ESI-TOF) calculated for $\text{C}_{17}\text{H}_{17}\text{BrO}_2$, m/z $[\text{M}+\text{K}]^+$: 371.0048922; experimental value: 371.004088.

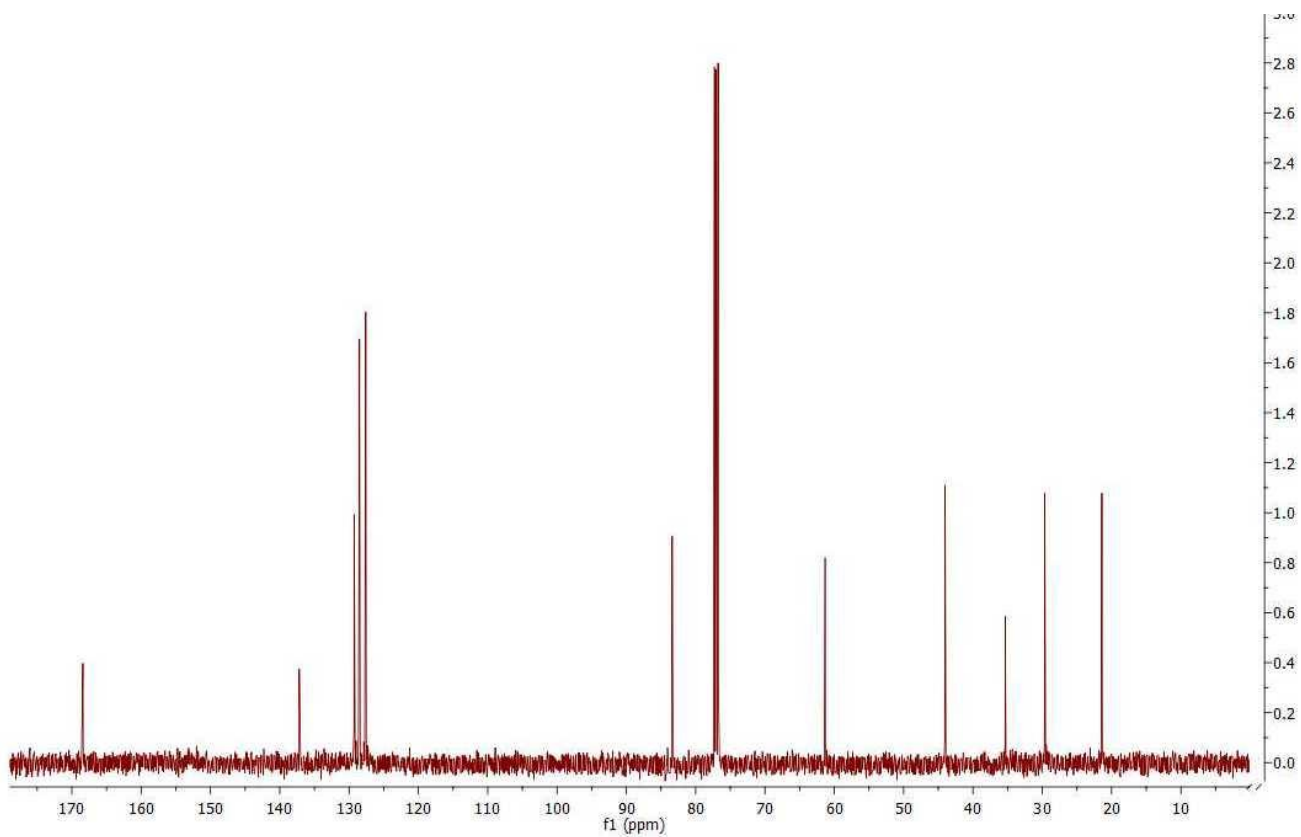


Compound 6b *trans*-5-bromo-4,4-dimethyl-6-phenyl-tetrahydro-2H-pyran-2-one

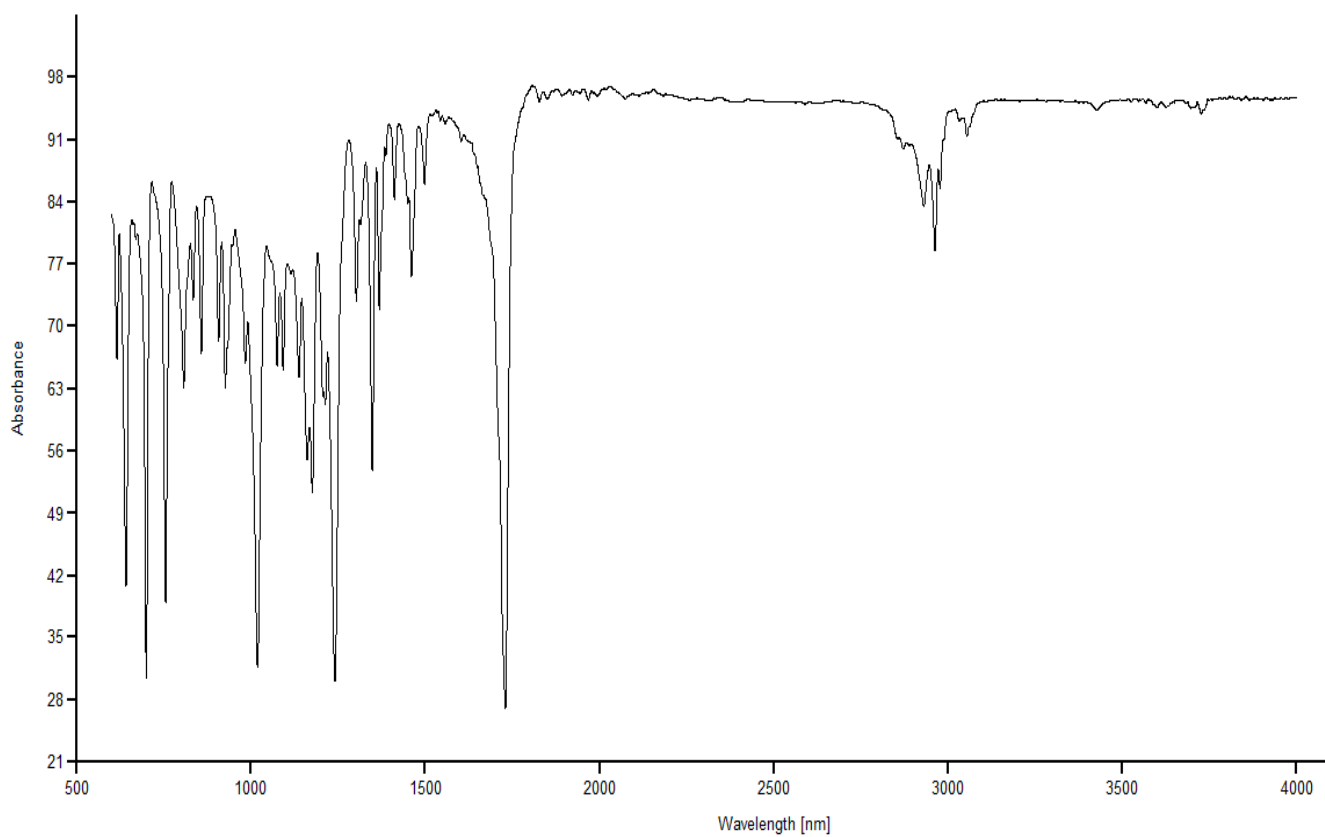
Yield 59%, colorless crystals, m.p. = 107–108 °C,



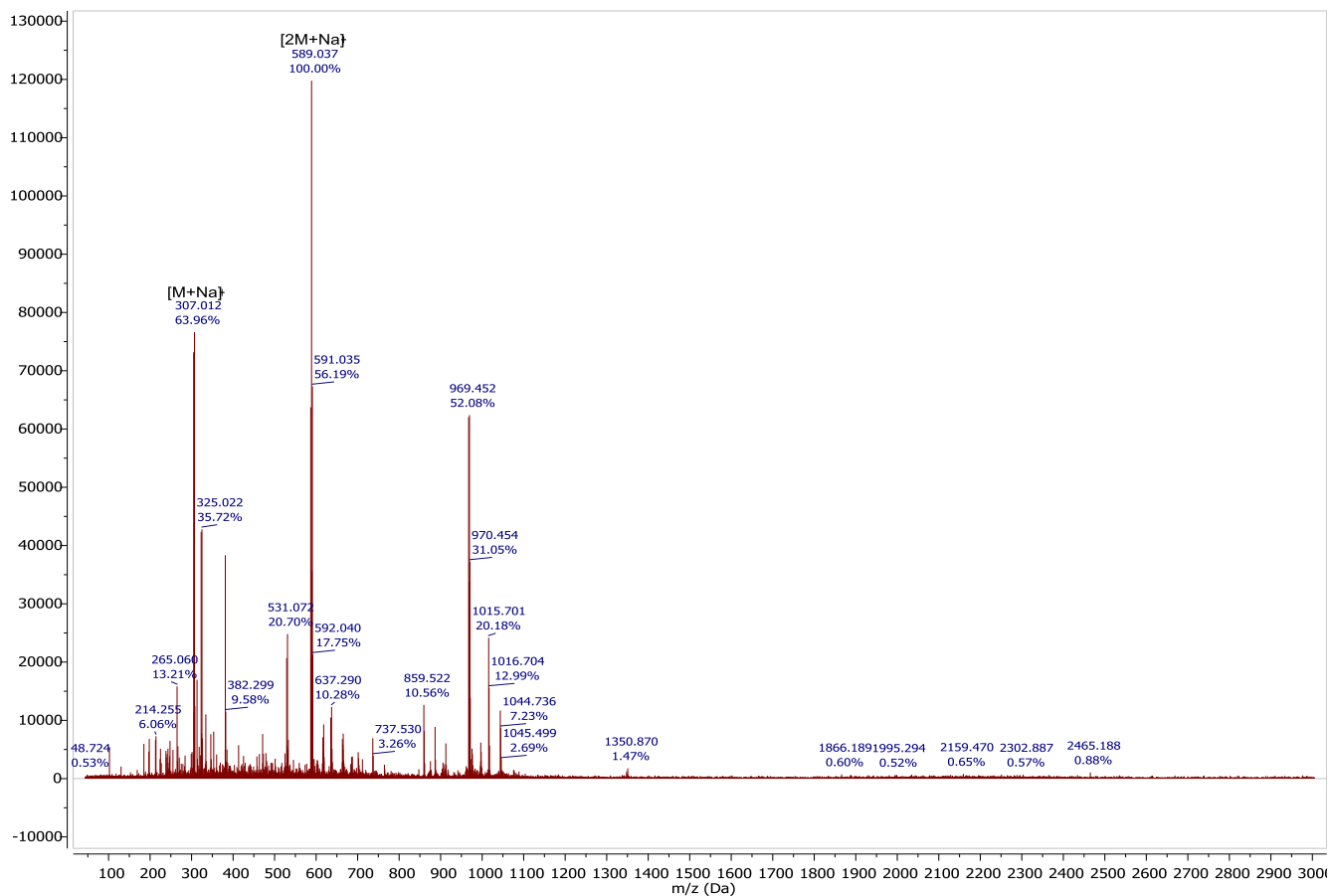
^1H NMR (CDCl_3 , 500 MHz), δ [ppm]: 1.20 (s, $\text{C}(\text{CH}_3)_2$, 3H), 1.28 (s, $\text{C}(\text{CH}_3)_2$, 3H), 2.61 (d, $J = 17.4$ Hz, CH_2 , 1H), 2.81 (d, $J = 17.4$ Hz, CH_2 , 1H), 4.11 (d, $J = 10.8$ Hz, CHBr , 1H), 5.40 (d, $J = 10.8$ Hz, CHAr , 1H), 7.35–7.42 (m, HAr , 5H).



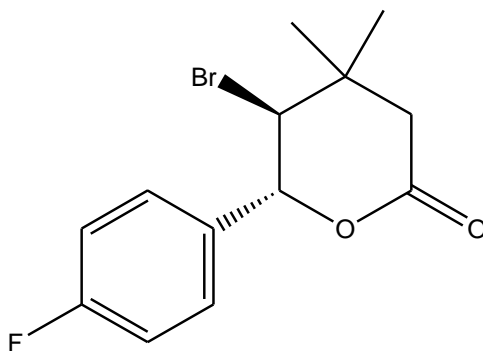
^{13}C NMR (125 Hz, CDCl_3) δ [ppm]: 21.40, 29.64, 35.53, 44.02, 61.33, 83.39, 168.40, 127.59, 128.50, 129.25, 137.19.



IR: 1730, 1462, 1340, 1235, 1180, 1017, 923, 860, 806, 755, 670, 640 cm^{-1} .

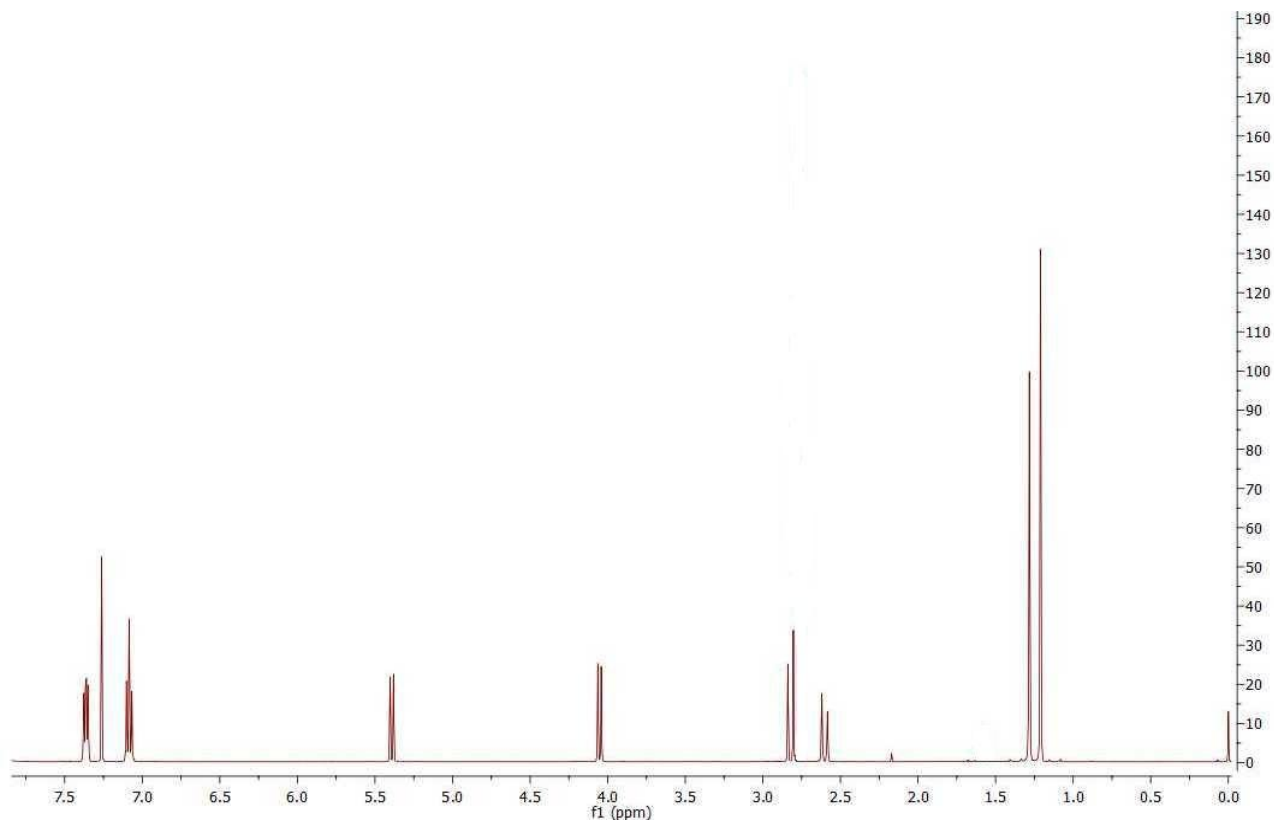


HR-MS (ESI-TOF) calculated for $C_{13}H_{15}BrO_2$, m/z $[M+Na]^+$: 305.015305; experimental value: 305.013815

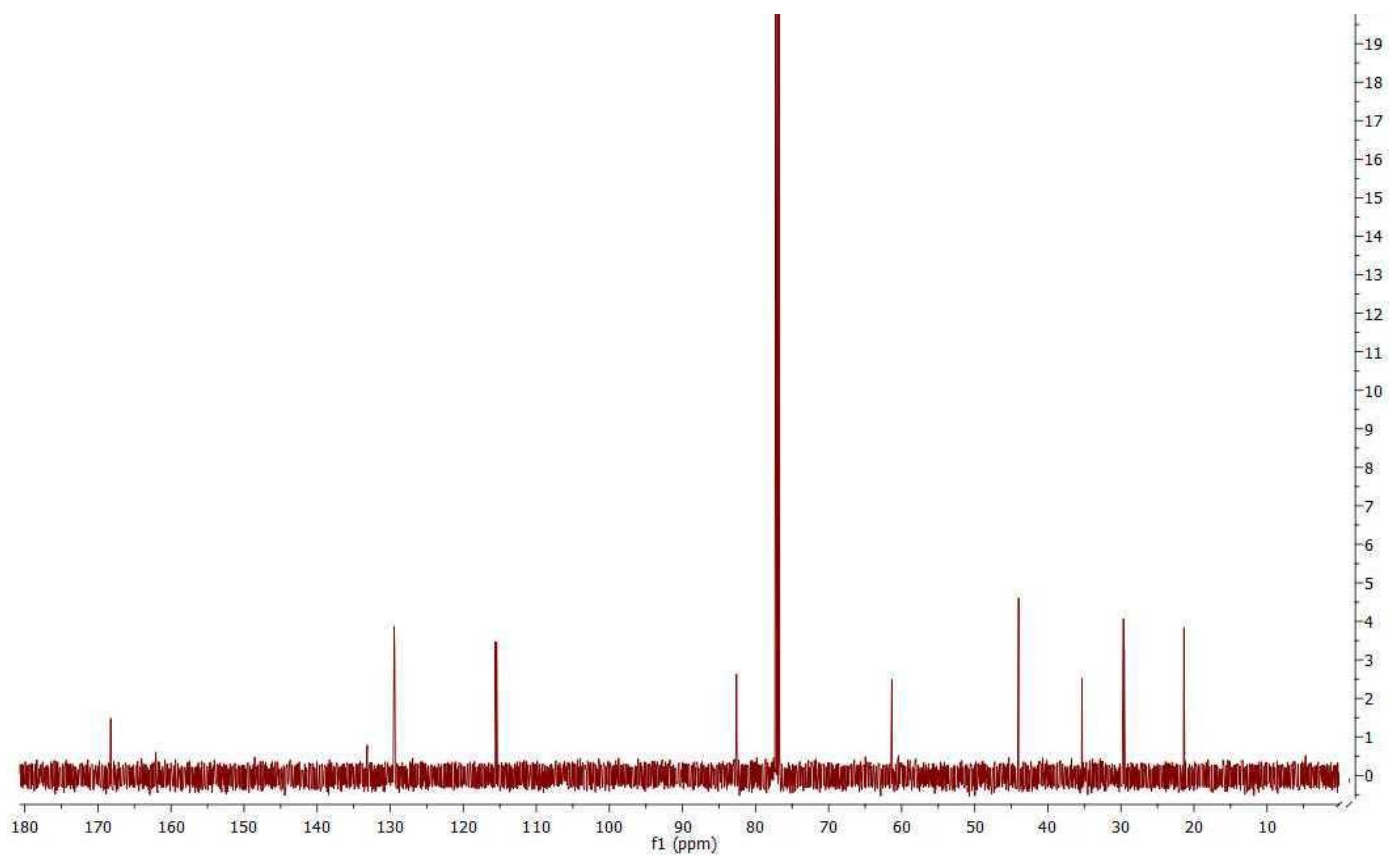


Compound 6c *trans*-5-bromo-6-(*p*-fluorophenyl)-4,4-dimethyltetrahydro-2H-pyran-2-one

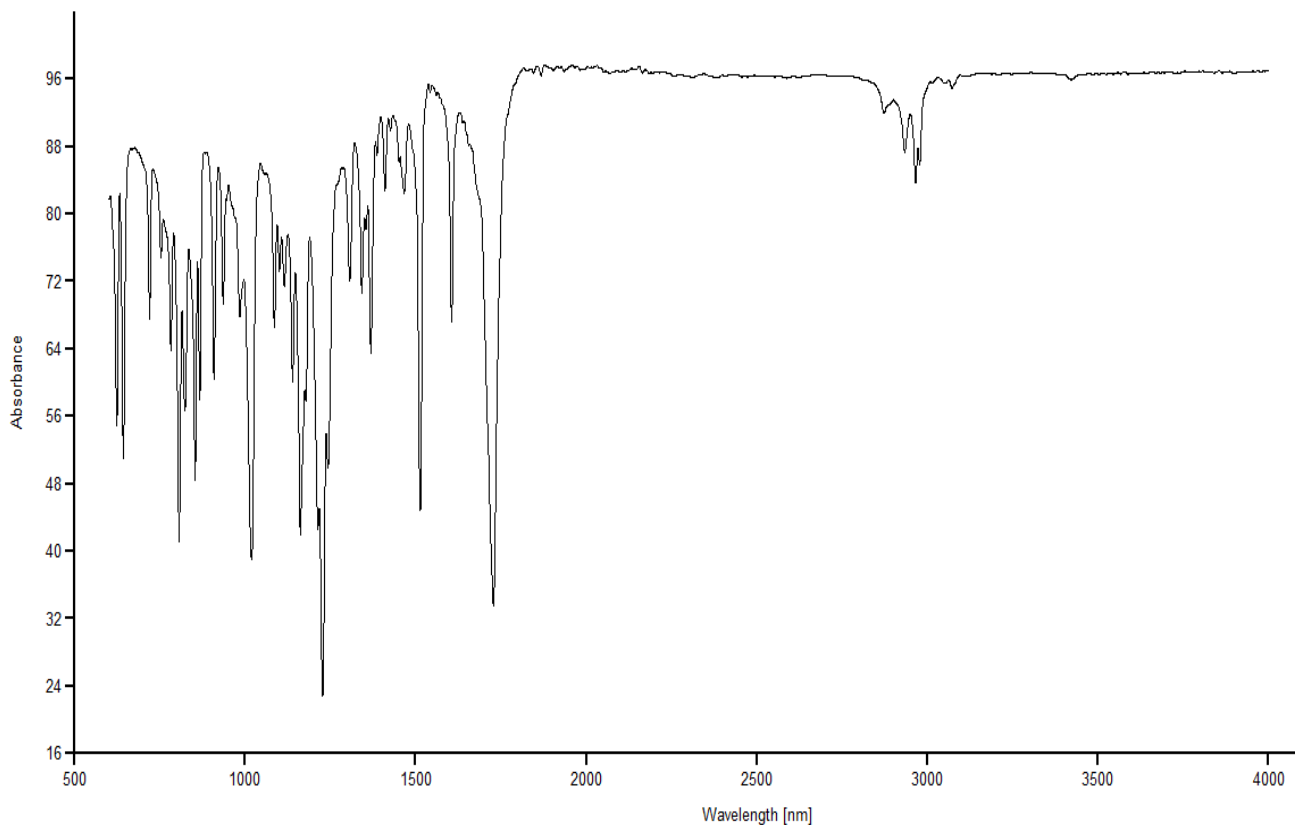
Yield 85%, colorless crystals, m.p. = 102–103 °C,



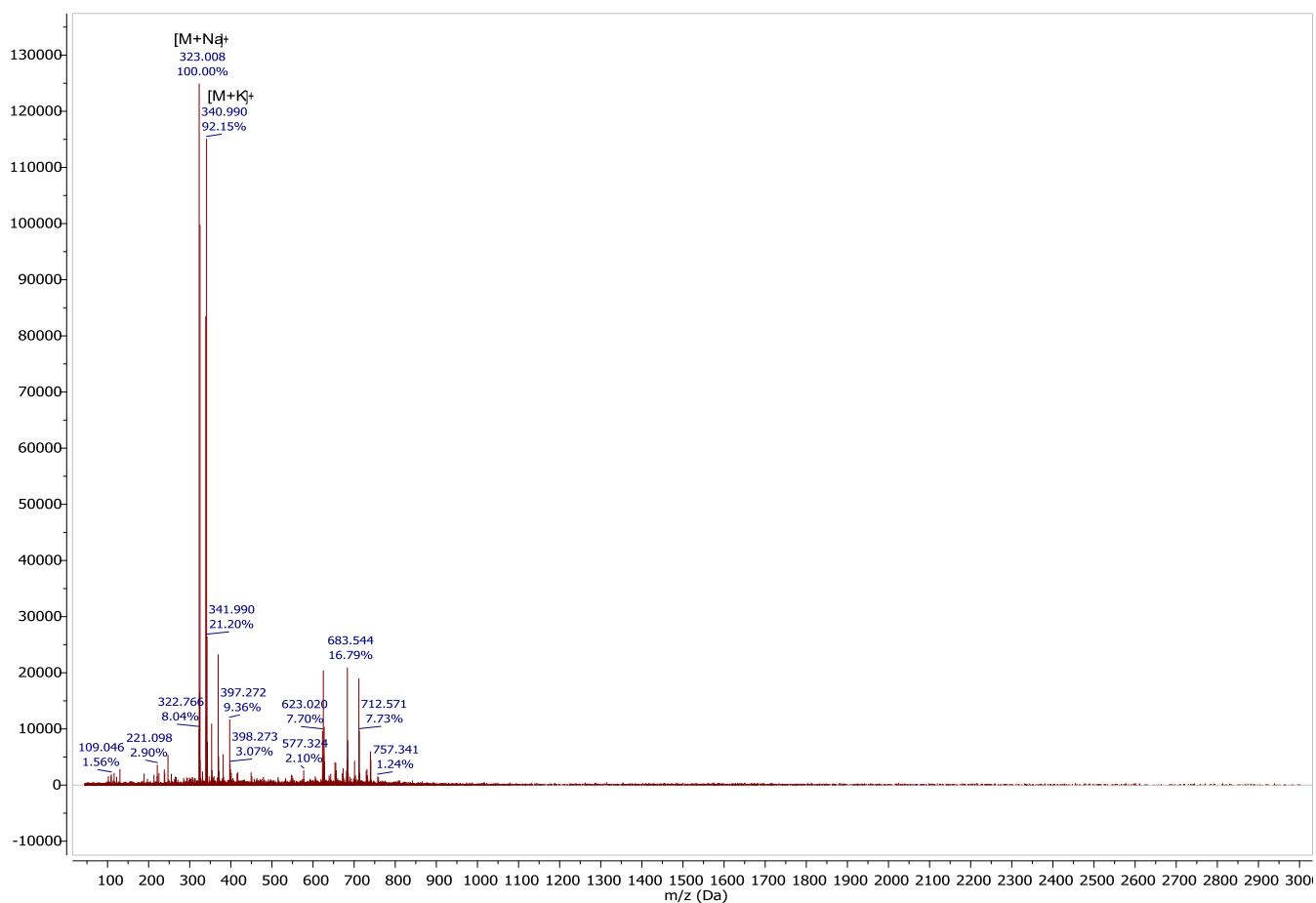
¹H NMR (CDCl₃, 500 MHz) δ [ppm]: 1.21 (s, C(CH₃)₂, 3H), 1.28 (s, C(CH₃)₂, 3H), 2.60 (d, *J* = 17.4 Hz, CH₂, 1H), 2.82 (d, *J* = 17.4 Hz, CH₂, 1H), 4.05 (d, *J* = 10.8 Hz, CHBr, 1H), 5.39 (d, *J* = 10.8 Hz, CHAr, 1H), 7.03–7.12 (m, HAr, 2H), 7.32–7.39 (m, HAr, 2H).



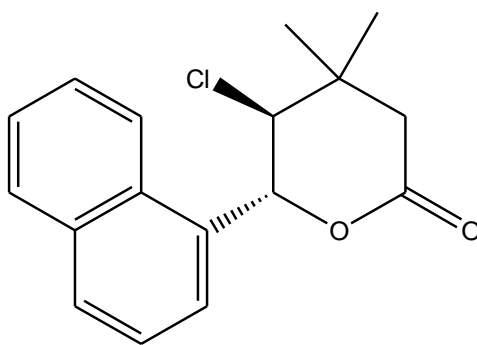
¹³C NMR (125Hz, CDCl₃) δ [ppm]: 21.38, 29.55, 35.34, 44.01, 61.35, 82.61, 115.52, 129.42, 133.12, 163.05, 168.25.



IR: 1726, 1510, 1360, 1230, 1160, 1020, 905, 855, 805, 650, 620 cm^{-1} .

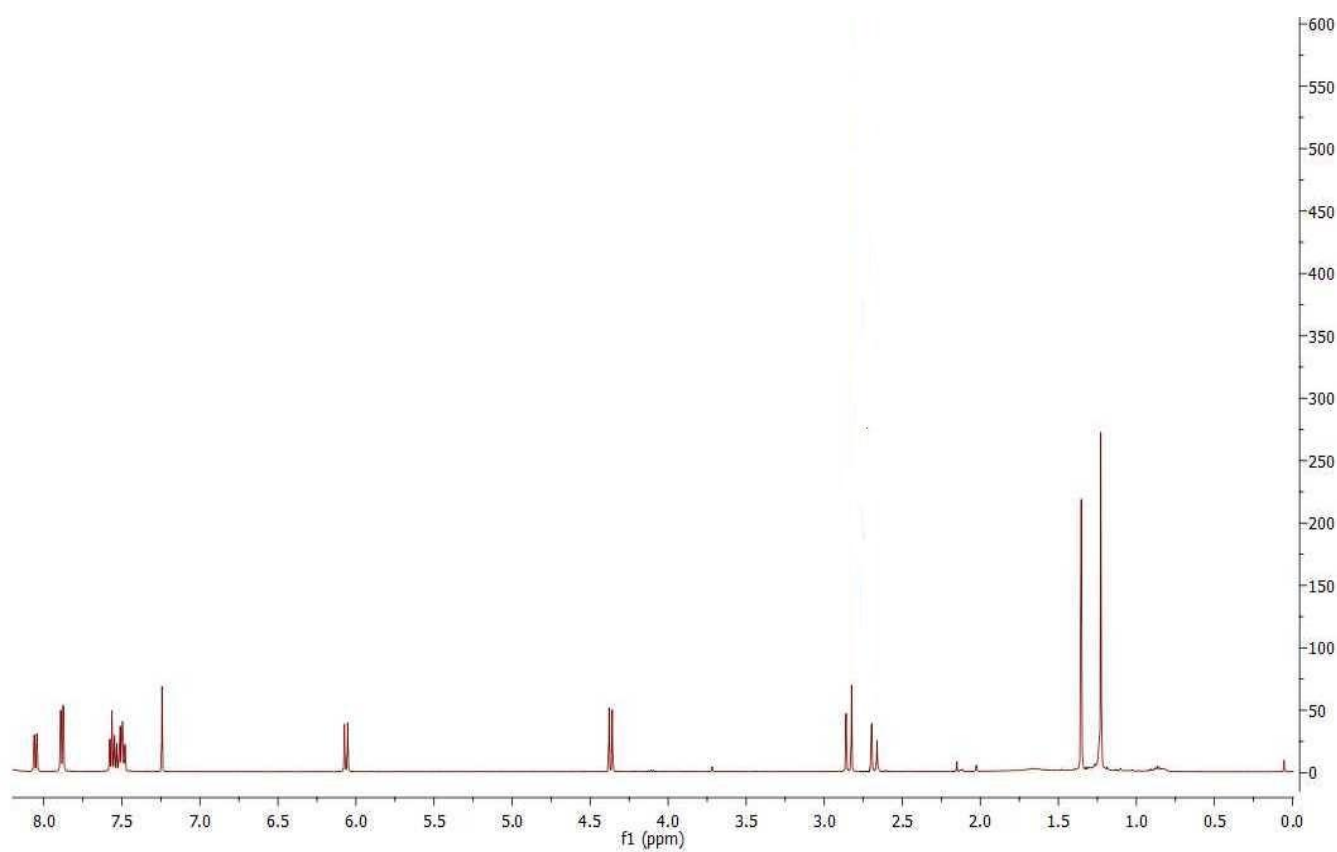


HR-MS (ESI-TOF) calculated for $\text{C}_{13}\text{H}_{14}\text{O}_2\text{BrF}$, m/z $[\text{M}+\text{Na}]^+$: 323,0058836; experimental value: 323.008034

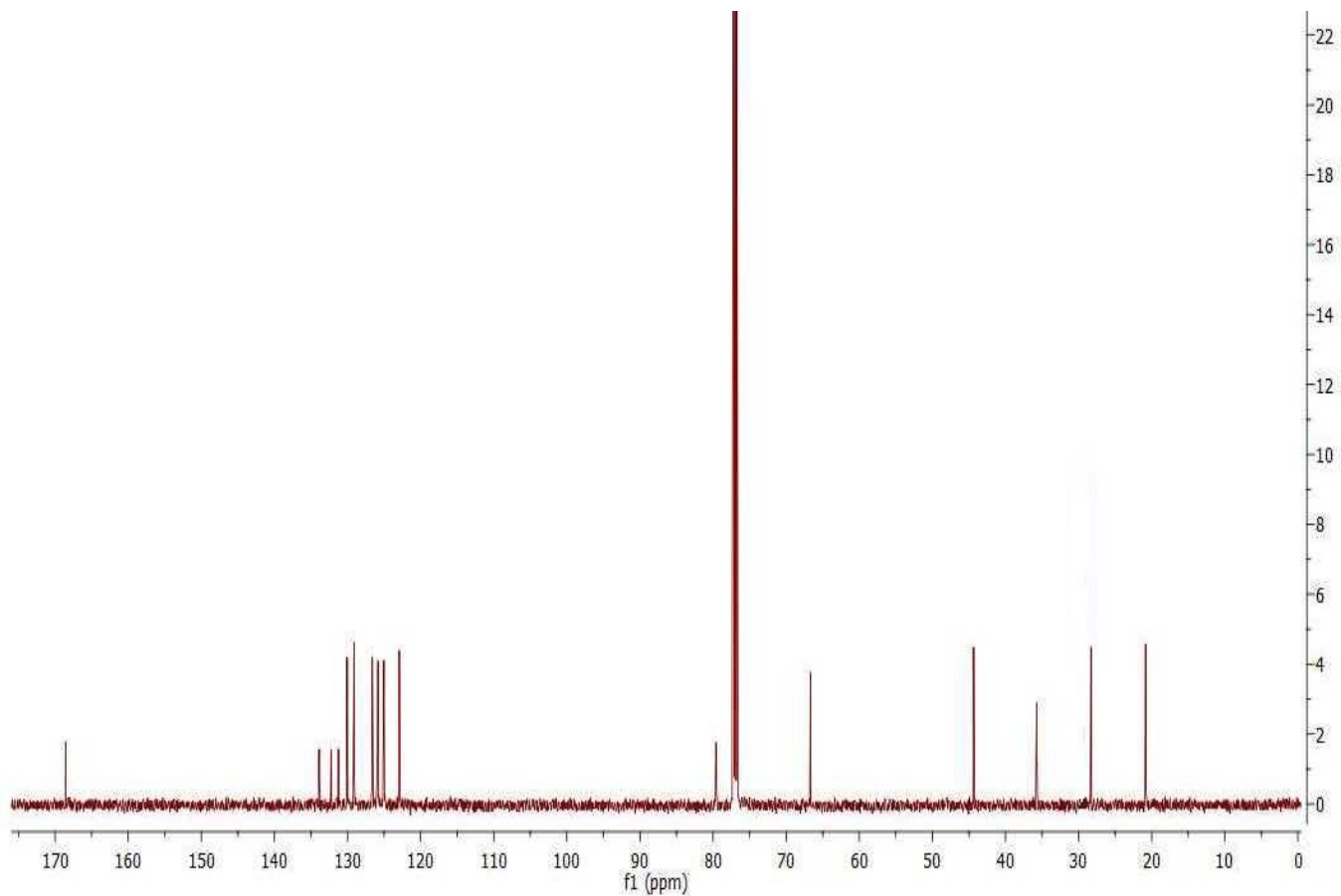


Compound 7a *trans*-5-chloro-4,4-dimethyl-6-(α -naphthyl)tetrahydro-2H-pyran-2-one

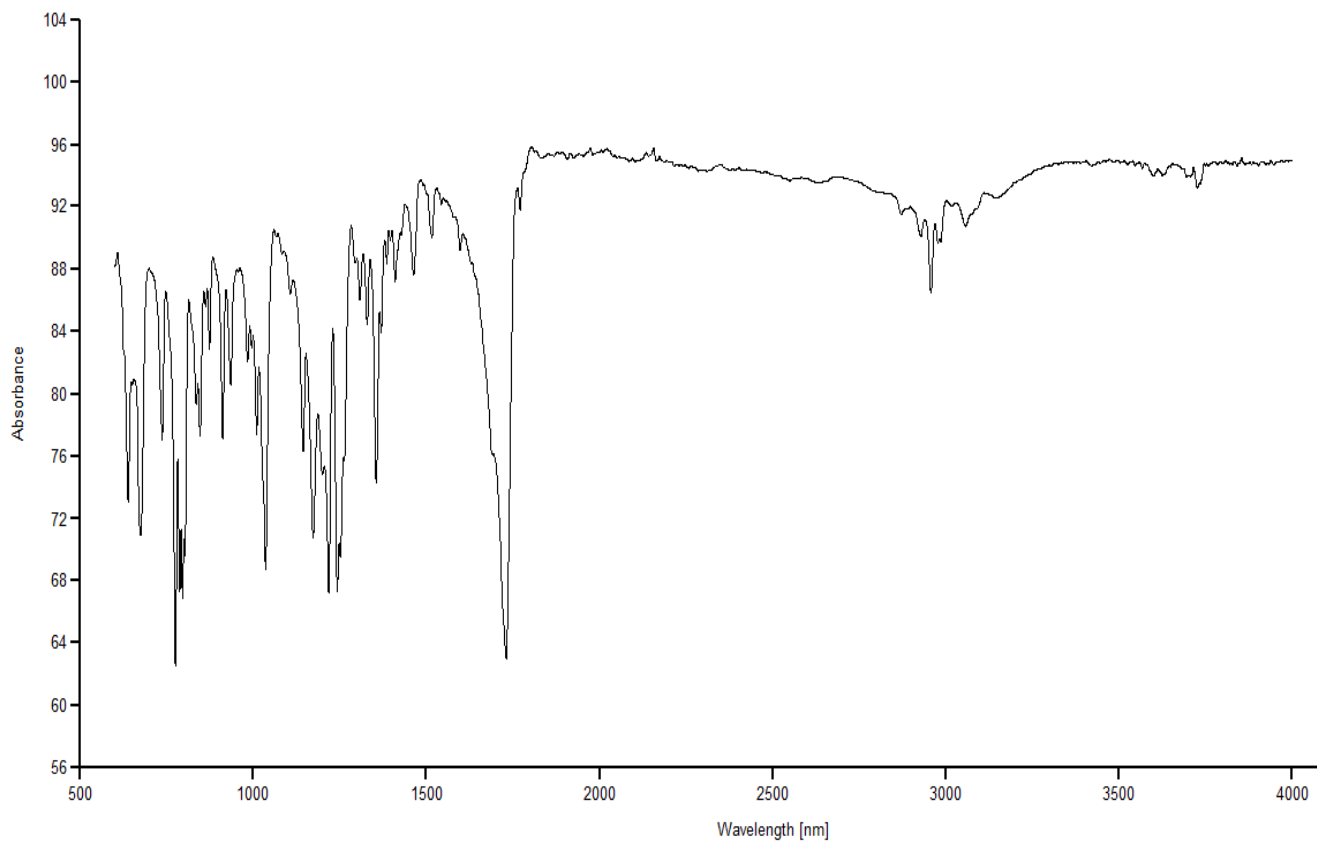
Yield 75%, colorless crystals, m.p. = 192–193 °C,



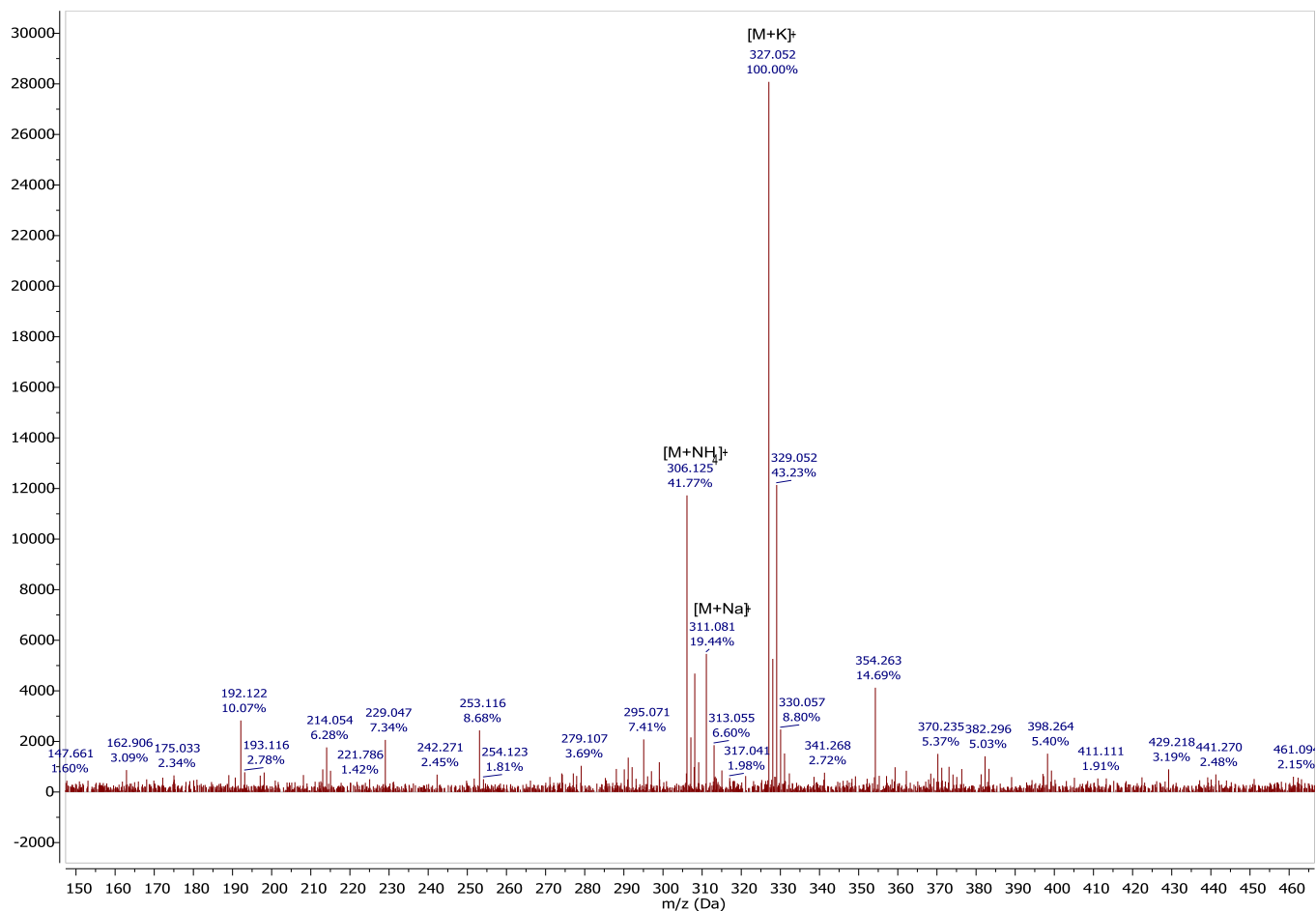
^1H NMR (CDCl_3 , 500 MHz), δ [ppm]: 1.23 (s, $\text{C}(\text{CH}_3)_2$, 3H), 1.35 (s, $\text{C}(\text{CH}_3)_2$, 3H), 2.68 (d, $J = 17.4$ Hz, CH_2 , 1H), 2.84 (d, $J = 17.4$ Hz, CH_2 , 1H), 4.37 (d, $J = 10.4$ Hz, CHCl , 1H), 6.06 (d, $J = 10.4$ Hz, CHAr , 1H), 7.47–7.60 (m, HAr , 4H), 7.88 (d, $J = 7.8$ Hz, HAr , 2H), 8.05 (d, $J = 8.5$ Hz, HAr , 1H).



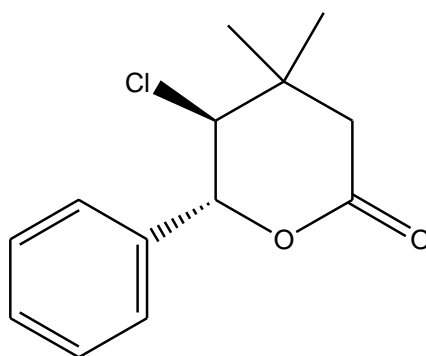
^{13}C NMR (125 Hz, CDCl_3) δ [ppm]: 20.83, 29.54, 35.72, 44.35, 66.68, 79.59, 122.92, 125.01, 125.84, 125.91, 126.61, 129.10, 130.06, 131.24, 132.24, 133.87, 168.56.



IR: 1730, 1350, 1240, 1216, 1170, 1140, 1035, 930, 910, 850, 790, 775, 730, 675, 640 cm^{-1} .

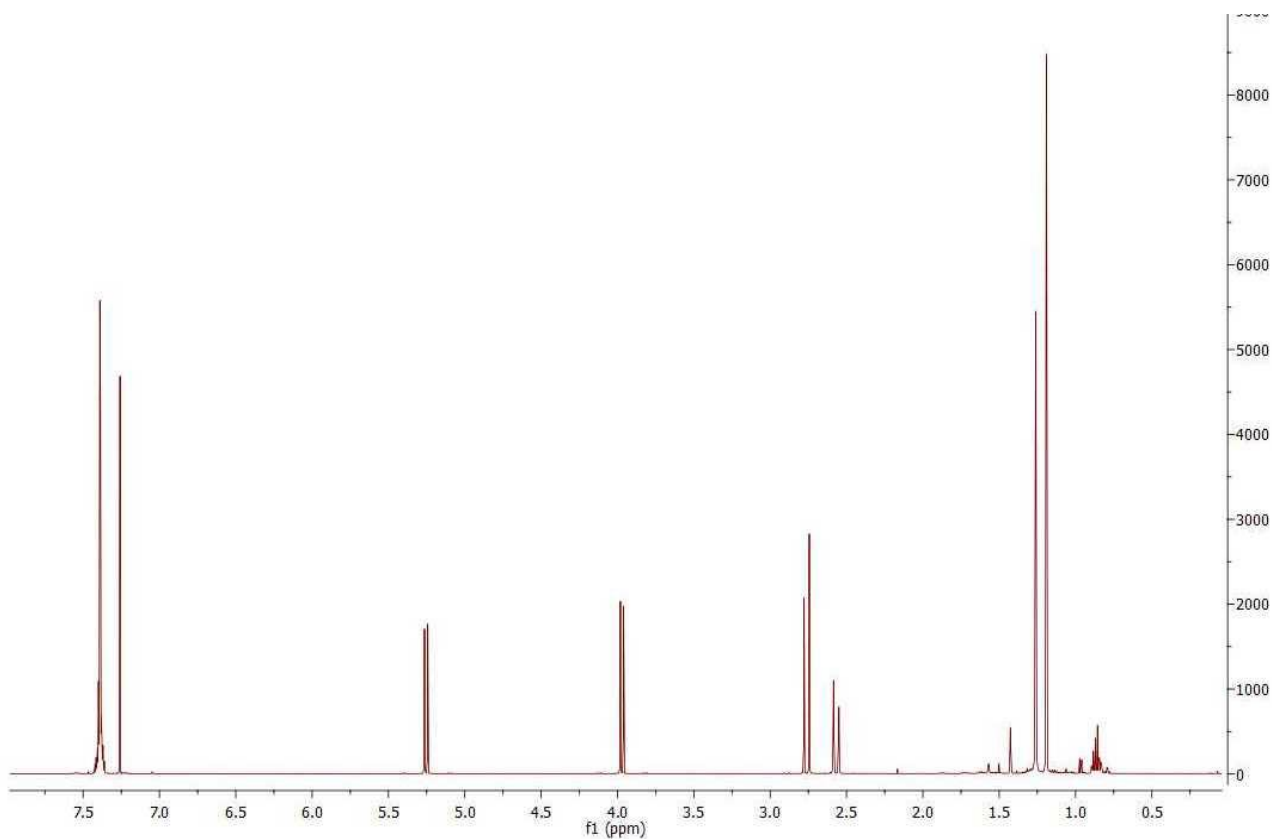


HR-MS (ESI-TOF) calculated for $C_{17}H_{17}ClO_2$, m/z $[M+Na]^+$: 311.0814712; experimental value: 311.080645.

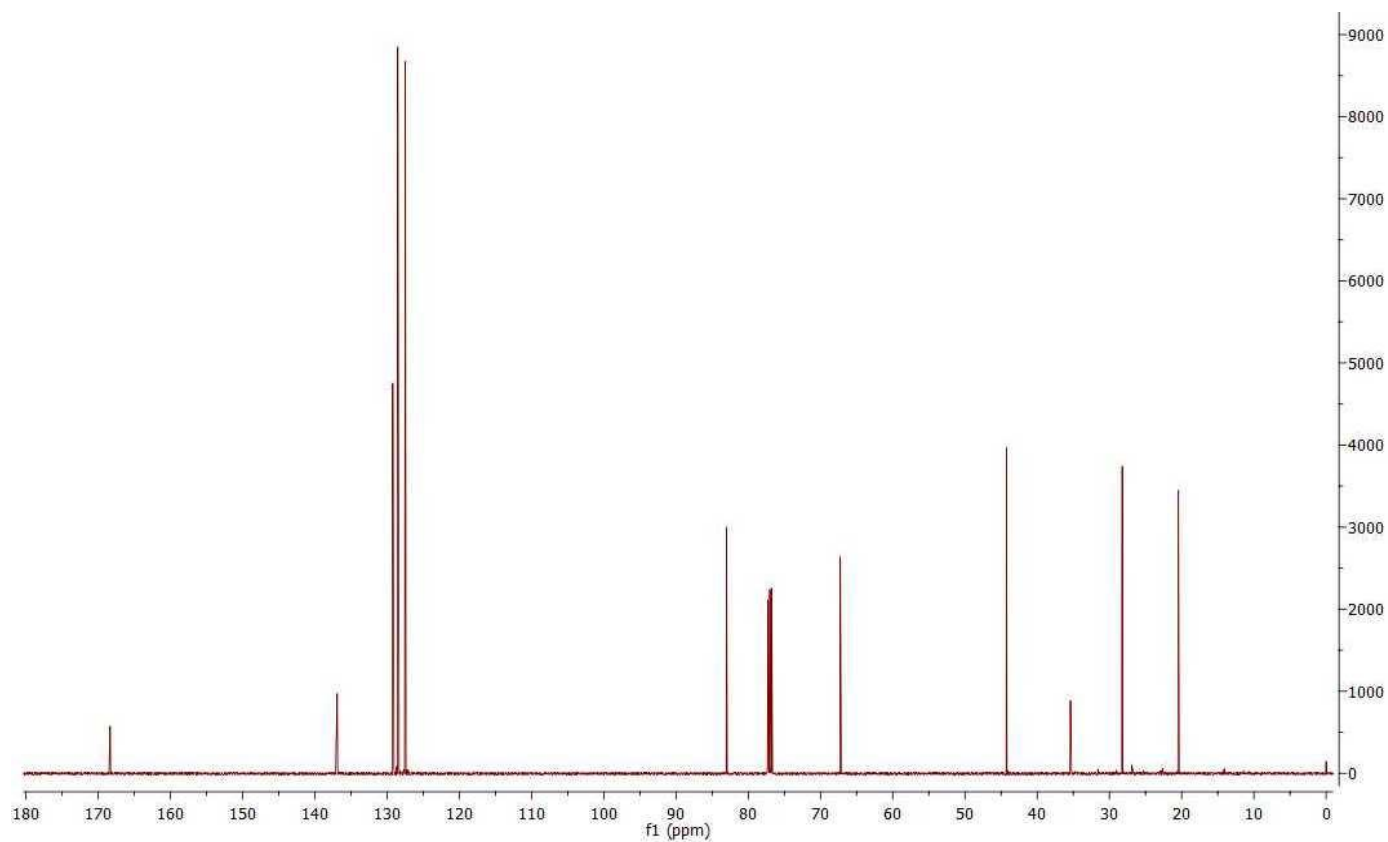


Compound 7b *trans*-5-chloro-4,4-dimethyl-6-phenyltetrahydro-2H-pyran-2-one

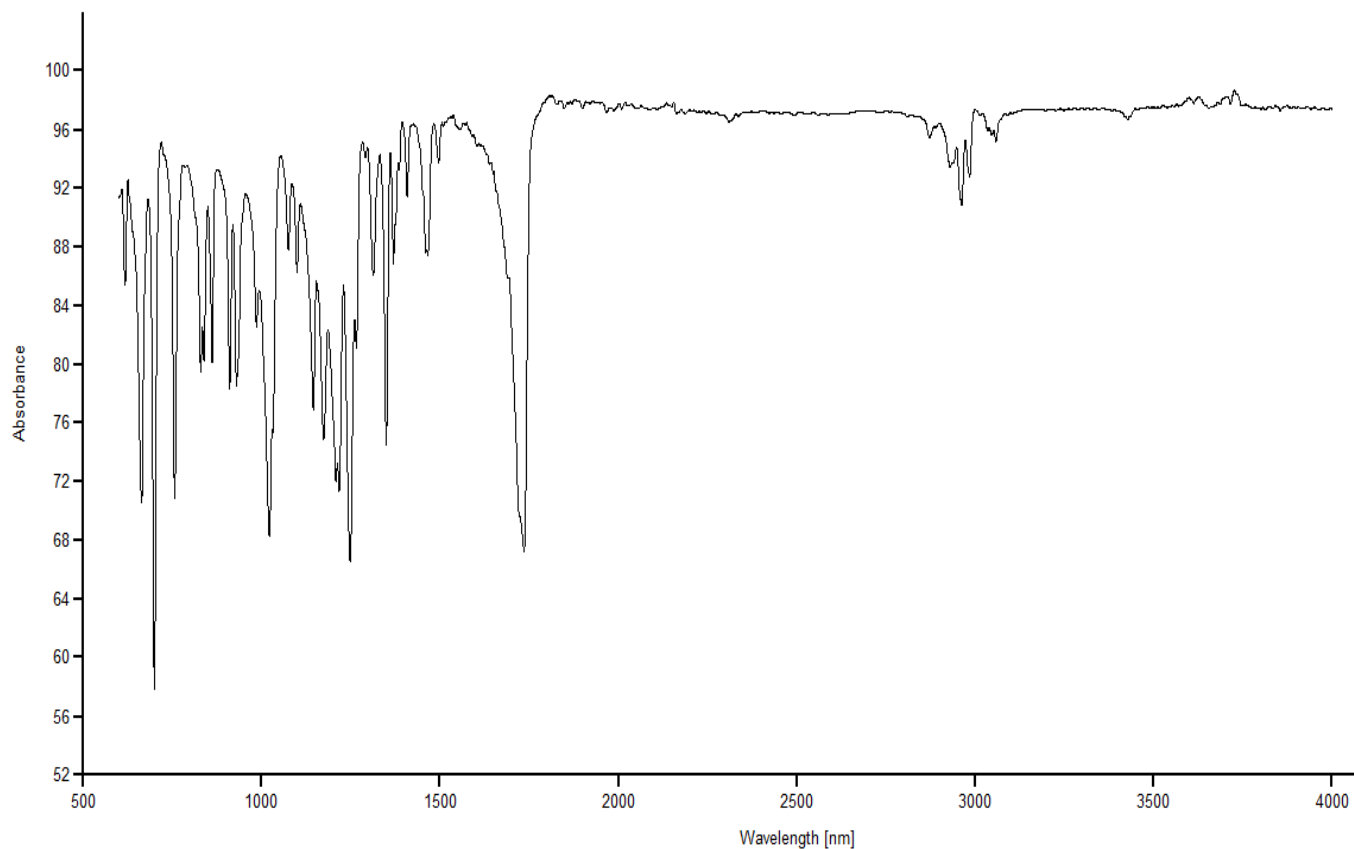
Yield 85%, colorless crystals, m.p. = 96-97 °C,



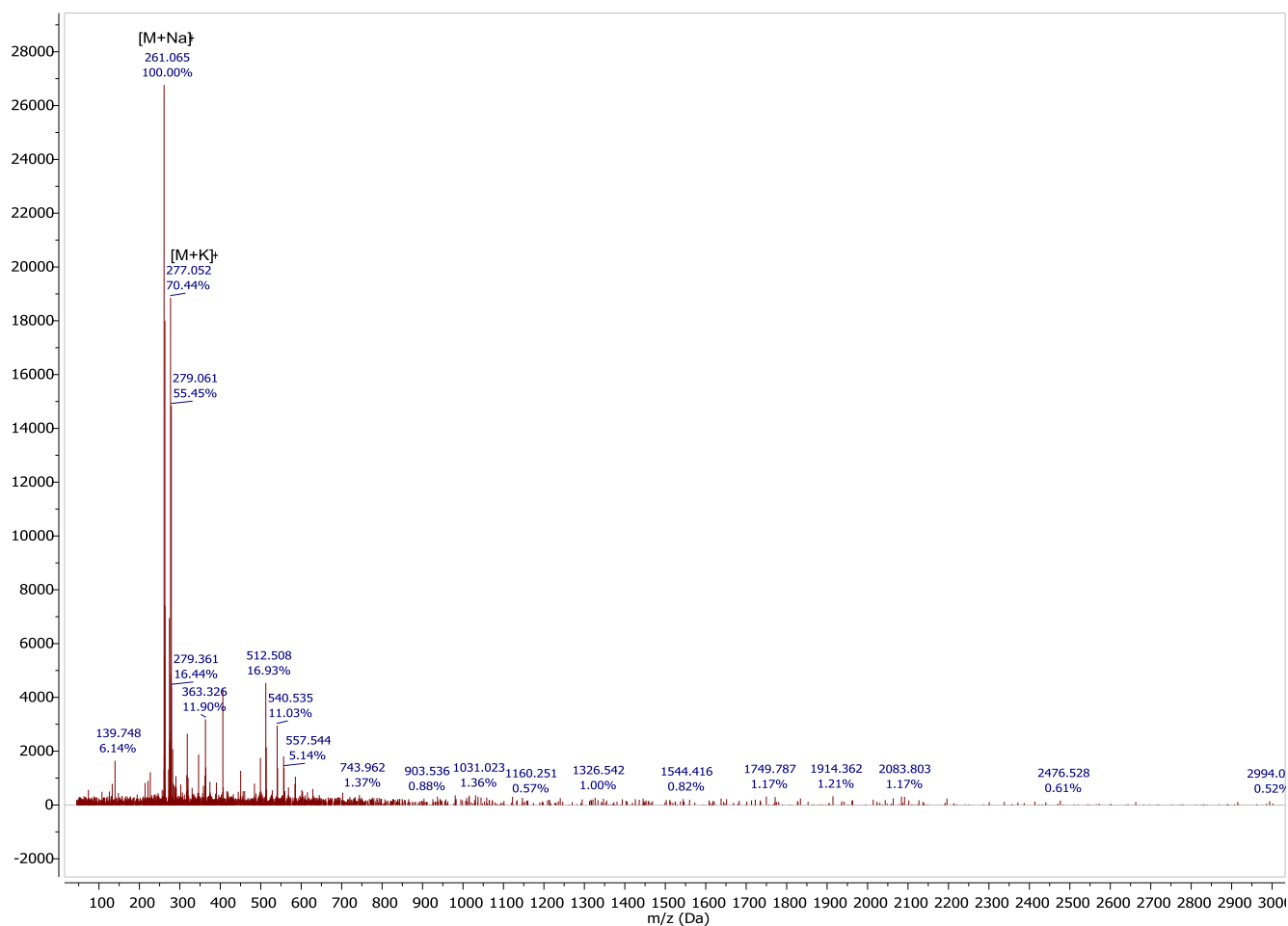
^1H NMR (CDCl_3 , 500 MHz), δ [ppm]: 1.19 (s, $\text{C}(\text{CH}_3)_2$, 3H), 1.26 (s, $\text{C}(\text{CH}_3)_2$, 3H), 2.57 (d, $J = 17.4$ Hz, CH_2 , 1H), 2.76 (d, $J = 17.4$ Hz, CH_2 , 1H), 3.97 (d, $J = 10.4$ Hz, CHCl , 1H), 5.25 (d, $J = 10.4$ Hz, CHAr , 1H), 7.33–7.44 (m, HAr , 5H).



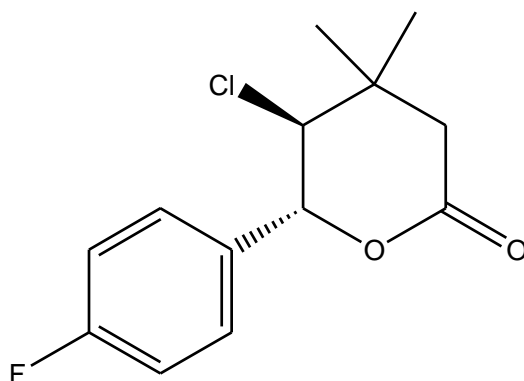
^{13}C NMR (125 Hz, CDCl_3) δ [ppm]: 20.45, 28.22, 35.39, 44.26, 67.26, 83.01, 127.48, 128.53, 129.22, 136.92, 168.35.



IR: 1733, 1460, 1350, 1250, 1215, 1174, 1143, 1020, 930, 910, 863, 830, 760, 700, 660 cm^{-1} .

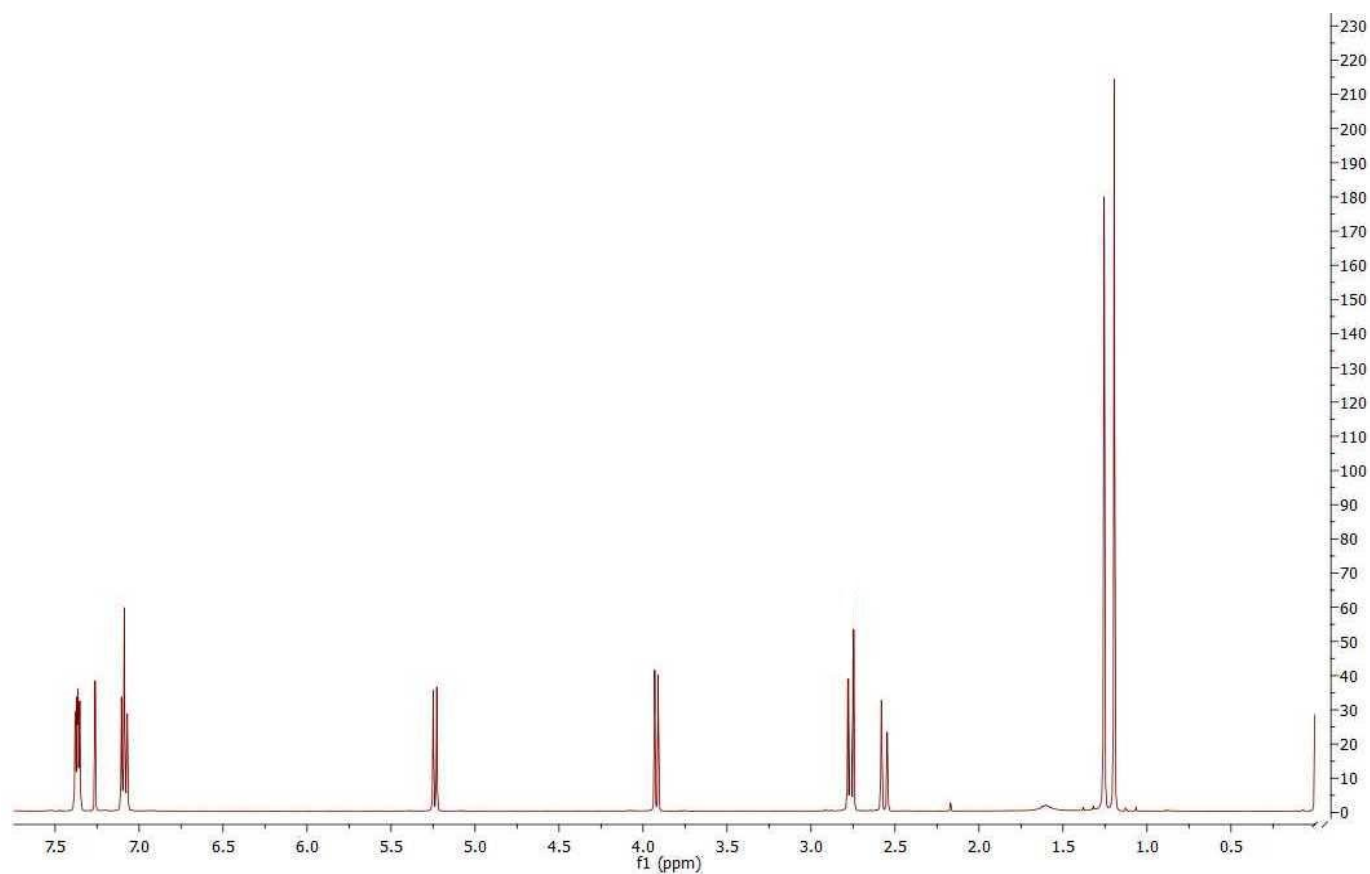


HR-MS (ESI-TOF) calculated for $\text{C}_{13}\text{H}_{15}\text{ClO}_2$, m/z $[\text{M}+\text{Na}]^+$: 261.065822; experimental value: 261.065139

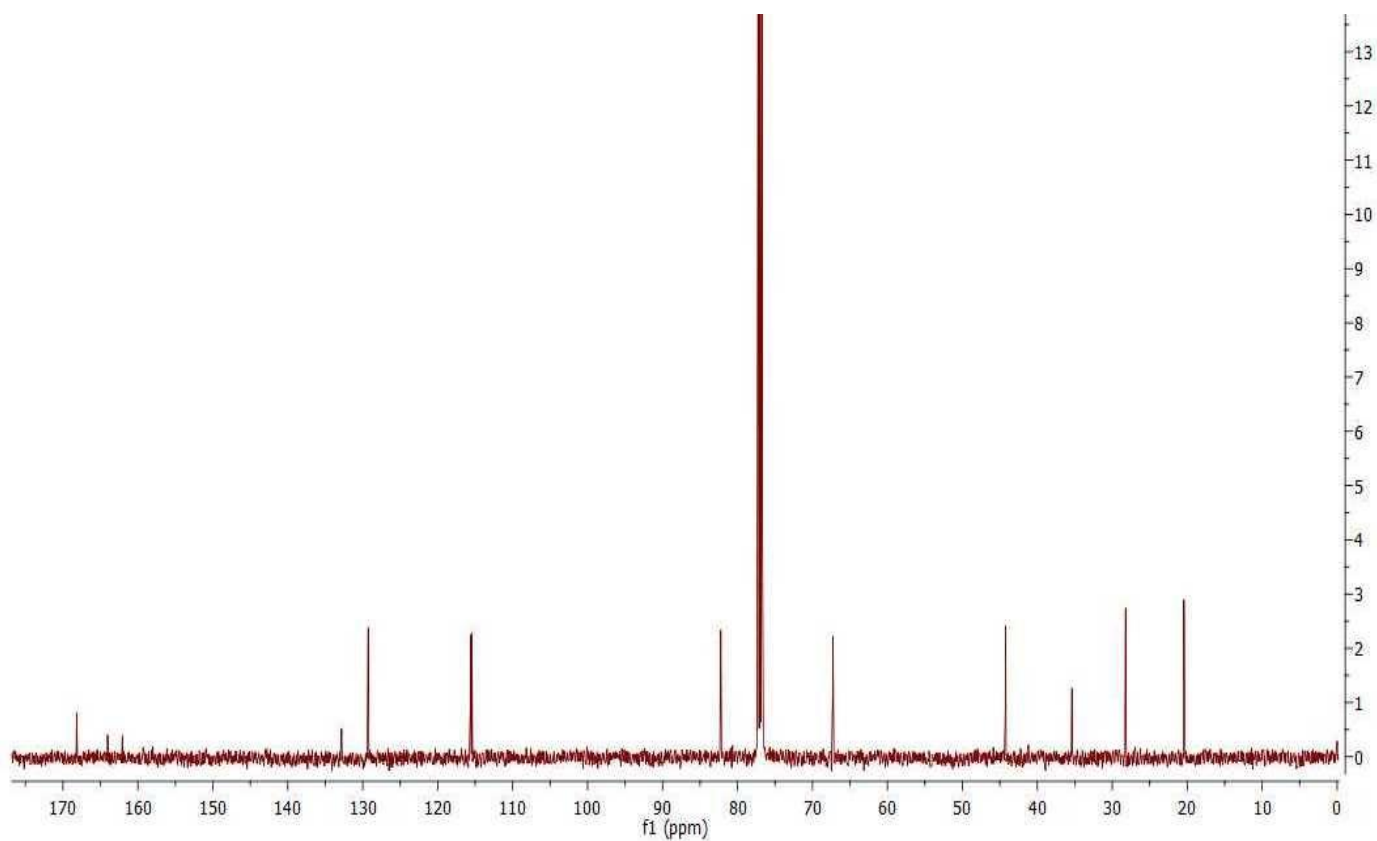


Compound 7c *trans*-5-chloro-6-(*p*-fluorophenyl)-4,4-dimethyltetrahydro-2H-pyran-2-one

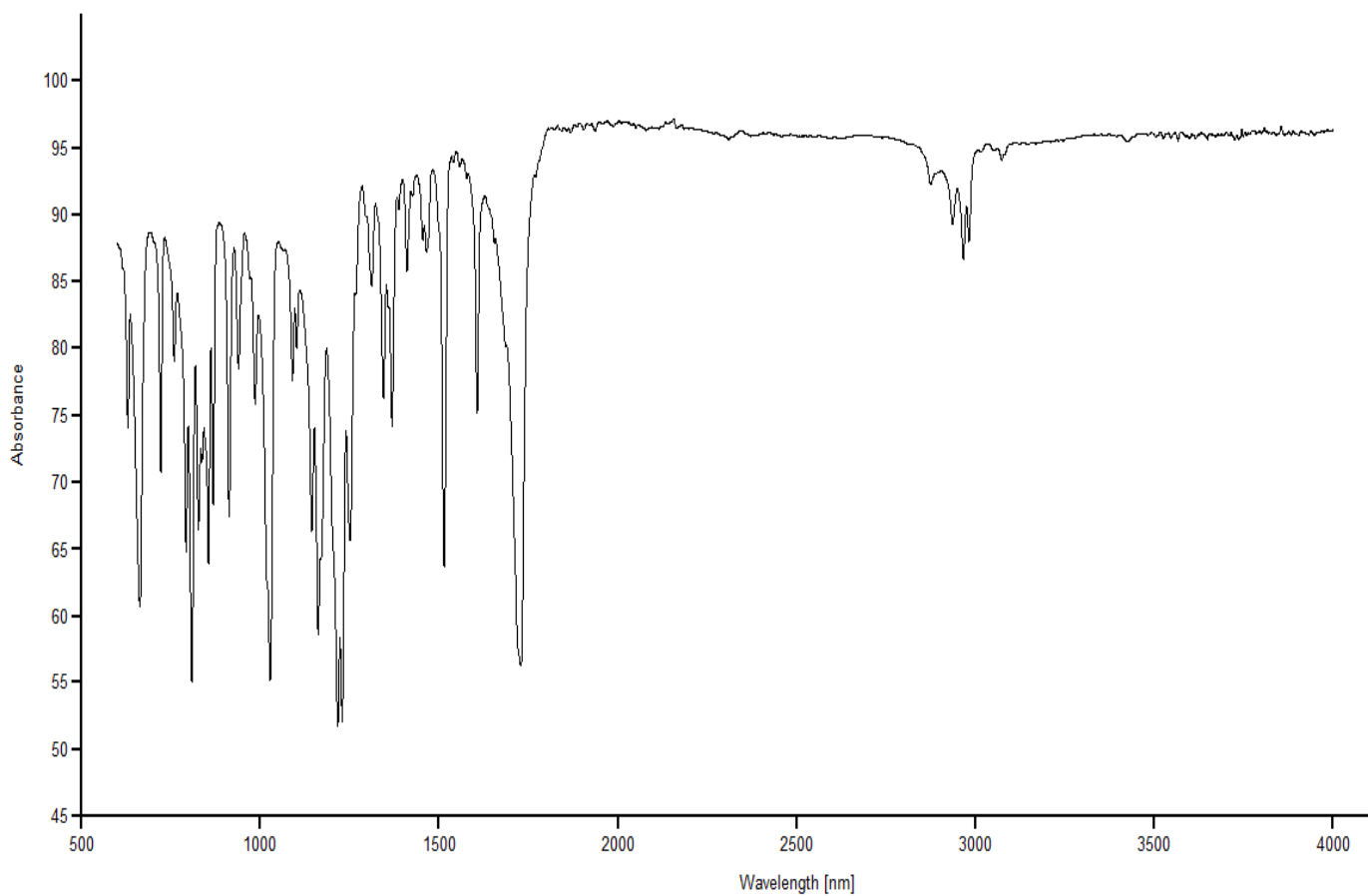
Yield 65%, colorless crystals, m.p. = 89-90 °C,



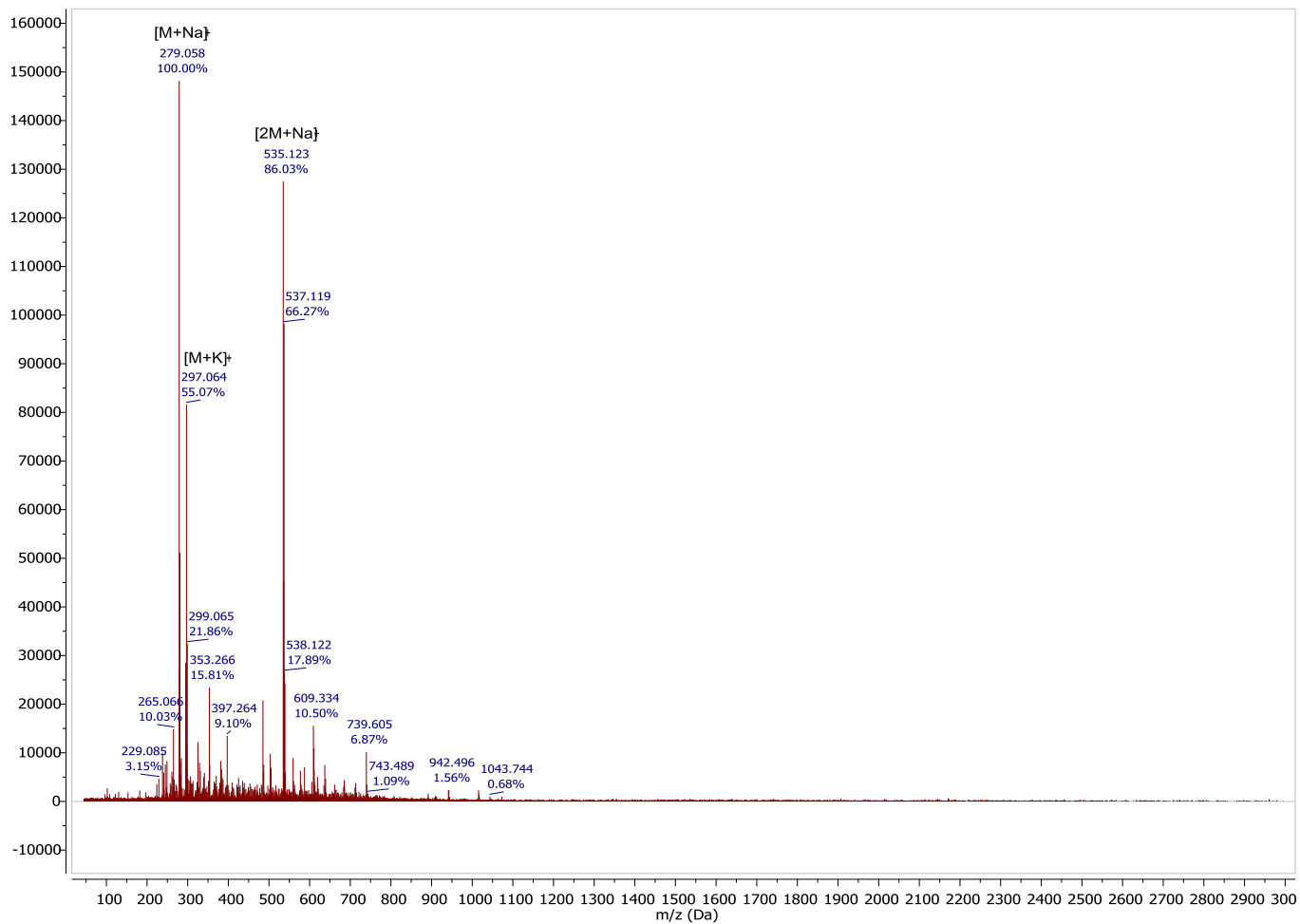
^1H NMR (CDCl_3 , 500 MHz) δ [ppm]: 1.19 (s, $\text{C}(\text{CH}_3)_2$, 3H), 1.26 (s, $\text{C}(\text{CH}_3)_2$, 3H), 2.56 (d, $J = 17.4$ Hz, CH_2 , 1H), 2.76 (d, $J = 17.4$ Hz, CH_2 , 1H), 3.92 (d, $J = 10.5$ Hz, CHCl , 1H), 5.24 (d, $J = 10.5$ Hz, CHAr , 1H), 6.98–7.14 (m, HAr , 2H), 7.31–7.42 (m, HAr , 2H).



^{13}C NMR (125Hz, CDCl_3) δ [ppm]: 20.43, 28.22, 35.38, 44.24, 67.28, 82.23, 115.53, 129.30, 132.80, 163.05, 168.17.



IR: 1730, 1605, 1515, 1370, 1340, 1250, 1230, 1160, 1025, 913, 850, 805, 722, 660, 625 cm^{-1} .



HR-MS (ESI-TOF) calculated for $C_{13}H_{14}ClFO_2$, m/z $[M+Na]^+$: 279.0564006; experimental value: 279.057500.