

Supplemental Material

Metabolic acid-catalyzed synthesis of cyclic carbonates from epoxides and CO₂

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General Information

Unless otherwise specified, all reagents and solvents are purchased from suppliers and used directly without further purification. Column chromatography was performed on silica gel (200-300 mesh) using petroleum ether/ethyl acetate as eluent. All ¹H NMR (400 MHz or 500MHz), ¹³C NMR (101 MHz or 126 MHz) were recorded on Bruker AVANCE II-400 or Bruker AVANCE III-500 spectrometers (in CDCl₃ with TMS as internal standard). Melting points were recorded on a Novel X-4 spectrometer. Infrared spectroscopies were recorded on Nicolet 6700 Fourier transform infrared spectrometer. Mass spectroscopies were recorded on the LTQ Orbitrap XL mass spectrometer.

General synthesis procedure

The reaction was carried out in a 25 ml high-pressure reactor. VCHO (20 mmol), HBO₂ (0.25 mmol), and TBAB (0.6 mmol) were added to the reactor at room temperature. The reactor was purged with argon gas three times, followed by two purges with carbon dioxide. Then, the reactor was pressurized to 14 bar and heated in an oil bath at 140 °C for 5 hours. After the completion of the reaction, the reactor was cooled to room temperature followed by gradual release of the pressure inside the reactor. The cyclic carbonate product was separated and purified by silica gel column chromatography. The structure of the product was determined by ¹H NMR, ¹³C NMR, IR and MS spectroscopy.

The characterization data of 2a-2s

The cyclic carbonates **2a-2s** are all known compounds, and the characterization data are consistent with previous reports.

5-Vinylhexahydrobenzo[d][1,3]dioxol-2-one (2a):¹ the ratio of two diastereoisomers is approximately 1:1, 93% yield, colorless oil, ¹H NMR (500 MHz, CDCl₃) δ 5.73 – 5.56 (m, 2H), 5.02 – 4.82 (m, 4H), 4.76 – 4.52 (m, 4H), 2.30 – 1.99 (m, 5H), 1.96 – 1.91 (m, 1H), 1.76 – 1.46 (m, 5H), 1.24-1.15 (m, 2H), 1.12 – 1.04 (m, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 155.1, 155.1, 141.2, 141.1, 114.0, 113.7, 76.0, 75.7, 75.6, 75.1, 36.1, 33.8, 33.4, 31.4, 26.6, 25.6, 25.5, 24.9. MS (ESI) calculated for C₉H₁₃O₃ [M+H]⁺: 169.09; found: 168.96. IR (KBr, cm⁻¹) ν = 3081, 2943, 2867, 1796, 1641, 1357, 1191, 1147, 1032, 918, 782, 732.

Hexahydrobenzo[d][1,3]dioxol-2-one (2b):¹ 63% yield, white solid, mp: 45 – 46 °C, ¹H NMR (400 MHz, CDCl₃) δ 4.66 (td, J = 3.7, 1.9 Hz, 2H), 1.91 – 1.80 (m, 4H), 1.66 – 1.49 (m, 2H), 1.48 – 1.23 (m, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 155.4, 75.8, 26.6, 19.0. MS (ESI) calculated for C₇H₁₁O₃ [M+H]⁺: 143.07; found: 142.94. IR (KBr, cm⁻¹) ν = 2945, 2869, 1800, 1453, 1352, 1308, 1252, 1208, 1166, 1137, 1103, 1030, 994, 953, 904, 856, 820, 782, 730, 706, 588, 560.

Tetrahydro-4H-cyclopenta[d][1,3]dioxol-2-one (2c):¹ 79% yield, white solid, mp: 42 – 43 °C, ¹H NMR (400 MHz, CDCl₃) δ 5.18 – 5.01 (m, 2H), 2.22 – 2.05 (m, 2H), 1.84 – 1.61 (m, 4H). ¹³C NMR (101 MHz, CDCl₃) δ 155.5, 82.0, 33.1, 21.5. MS (ESI) calculated for C₆H₉O₃ [M+H]⁺: 129.05; found: 128.98. IR (KBr, cm⁻¹) ν = 3020, 2986, 2940,

2884, 2858, 2544, 2349, 1801, 1544, 1474, 1445, 1375, 1334, 1182, 1112, 1046, 928, 902, 863, 771, 698, 597, 579, 537.

Tetrahydrofuro[3,4-d][1,3]dioxol-2-one (2d):¹ 86% yield, white solid, mp: 86 – 87 °C, ¹H NMR (400 MHz, CDCl₃) δ 5.22 – 5.18 (m, 2H), 4.28 – 4.20 (m, 2H), 3.57 – 3.53 (m, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 154.6, 80.3, 73.0. MS (ESI) calculated for C₅H₁₀NO₄ [M+NH₄]⁺: 148.06; found: 148.02. IR (KBr, cm⁻¹) ν = 3052, 3007, 2928, 2872, 1785, 1546, 1471, 1370, 1279, 1242, 1170, 1110, 1050, 928, 900, 842, 772, 689, 600, 433.

4-Methyl-1,3-dioxolan-2-one (2e):² 88% yield, colorless oil, ¹H NMR (400 MHz, CDCl₃) δ 4.87 – 4.79 (m, 1H), 4.55 – 4.51 (m, 1H), 4.01 – 3.97 (m, 1H), 1.45 (d, J = 6.3 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 155.2, 73.7, 70.7, 19.3. MS (ESI) calculated for C₄H₇O₃ [M+H]⁺: 103.04; found: 102.93. IR (KBr, cm⁻¹) ν = 2992, 1790, 1483, 1388, 1352, 1184, 1119, 1074, 1052, 954, 850, 776, 712.

4-Ethyl-1,3-dioxolan-2-one (2f):² 88% yield, colorless oil, ¹H NMR (400 MHz, CDCl₃) δ 4.69 – 4.49 (m, 1H), 4.51 (t, J = 8.1 Hz, 1H), 4.09 – 4.05 (m, 1H), 1.91 – 1.69 (m, 2H), 1.02 (t, J = 7.5 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 155.2, 78.1, 69.1, 26.8, 8.4. MS (ESI) calculated for C₅H₉O₃ [M+H]⁺: 117.05; found: 116.94. IR (KBr, cm⁻¹) ν = 2976, 2942, 2886, 1793, 1484, 1396, 1377, 1302, 1177, 1111, 1061, 981, 776, 718.

4-Vinyl-1,3-dioxolan-2-one (2g):³ 70% yield, yellow oil, ¹H NMR (400 MHz, CDCl₃) δ 5.93 – 5.84 (m, 1H), 5.52 – 5.47 (m, 1H), 5.44 – 5.41 (m, 1H), 5.15 – 5.09 (m, 1H), 4.61 – 4.57 (m, 1H), 4.16 – 4.12 (m, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 154.9, 132.2, 121.1, 77.4, 69.1. MS (ESI) calculated for C₅H₇O₃ [M+H]⁺: 115.04; found: 114.86. IR (KBr, cm⁻¹) ν = 2993, 2923, 1804, 1482, 1434, 1386, 1328, 1173, 1073, 991, 945, 772, 724.

4-(Isopropoxymethyl)-1,3-dioxolan-2-one (2h):² 99% yield, yellow oil, ¹H NMR (400 MHz, CDCl₃) δ 4.81 – 4.74 (m, 1H), 4.47 (t, J = 8.3 Hz, 1H), 4.36 (dd, J = 8.3, 6.0 Hz, 1H), 3.67 – 3.56 (m, 3H), 1.17 – 1.10 (m, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 155.2, 75.4, 72.8, 67.1, 66.4, 21.8, 21.7. MS (ESI) calculated for C₇H₁₃O₄ [M+H]⁺: 161.08; found: 160.96. IR (KBr, cm⁻¹) ν = 2975, 2932, 2874, 1800, 1479, 1392, 1337, 1173, 1085, 920, 775, 714.

4-(Butoxymethyl)-1,3-dioxolan-2-one (2i):⁴ 81% yield, yellow oil, ¹H NMR (400 MHz, CDCl₃) δ 4.82 – 4.76 (m, 1H), 4.47 (t, J = 8.3 Hz, 1H), 4.37 (dd, J = 8.3, 6.0 Hz, 1H), 3.69 – 3.54 (m, 2H), 3.53 – 3.45 (m, 2H), 1.55 – 1.50 (m, 2H), 1.42 – 1.28 (m, 2H), 0.89 (t, J = 7.4 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 155.1, 75.3, 71.7, 69.6, 66.3, 31.5, 19.1, 13.8. MS (ESI) calculated for C₈H₁₅O₄ [M+H]⁺: 175.10; found: 175.03. IR (KBr, cm⁻¹) ν = 2960, 2935, 2873, 1797, 1481, 1395, 1362, 1175, 1136, 1103, 1052, 957, 849, 774, 714.

4-(tert-Butoxymethyl)-1,3-dioxolan-2-one (2j):⁵ 86% yield, colorless oil, ¹H NMR (400 MHz, CDCl₃) δ 4.77 – 4.71 (m, 1H), 4.44 (t, J = 8.3 Hz, 1H), 4.33 (dd, J = 8.3, 5.8 Hz, 1H), 3.58 (dd, J = 10.4, 4.0 Hz, 1H), 3.48 (dd, J = 10.4, 3.6 Hz, 1H), 1.15 (s, 9H). ¹³C NMR (101 MHz, CDCl₃) δ 155.3, 75.3, 73.7, 66.5, 61.2, 27.2. MS (ESI) calculated for C₈H₁₅O₄ [M+H]⁺: 175.10; found: 175.01. IR (KBr, cm⁻¹) ν = 2977, 2935, 2874, 1796, 1479, 1391, 1366, 1174, 1103, 1055, 1014, 884, 771, 713.

4-((Allyloxy)methyl)-1,3-dioxolan-2-one (2k):² 93% yield, colorless oil, ¹H NMR (400 MHz, CDCl₃) δ 5.85 – 5.76 (m, 1H), 5.26 – 5.11 (m, 2H), 4.81 – 4.76 (m, 1H), 4.45 (t, J = 8.4 Hz, 1H), 4.32 (dd, J = 8.4, 6.0 Hz, 1H), 4.01 – 3.97 (m, 2H), 3.64 (dd, J = 11.2, 3.2 Hz, 2H), 3.54 (dd, J = 11.2, 3.6 Hz, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 155.1, 133.8, 117.6, 75.3, 72.4, 68.9, 66.2. MS (ESI) calculated for C₇H₁₁O₄ [M+H]⁺: 159.07; found: 159.00. IR (KBr, cm⁻¹) ν =

2987, 2922, 2866, 1793, 1480, 1394, 1361, 1173, 1105, 1049, 999, 931, 774, 714.

4-(Chloromethyl)-1,3-dioxolan-2-one (2l):⁶ 88% yield, colorless oil, ^1H NMR (400 MHz, CDCl_3) δ 4.50 (d, $J = 8.8$ Hz, 1H), 4.15 (d, $J = 8.8$ Hz, 1H), 3.72 (d, $J = 11.8$ Hz, 1H), 3.59 (d, $J = 11.8$ Hz, 1H), 1.62 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 154.0, 81.9, 72.0, 48.6, 23.0. MS (ESI) calculated for $\text{C}_5\text{H}_8\text{ClO}_3$ [M+H] $^+$: 151.02; found: 150.96. IR (KBr, cm^{-1}) ν = 2986, 1805, 1453, 1481, 1393, 1298, 1159, 1065, 956, 769, 712, 584, 513.

4,4-Dimethyl-1,3-dioxolan-2-one (2m):² 83% yield, colorless oil, ^1H NMR (400 MHz, CDCl_3) δ 4.17 (s, 2H), 1.54 (s, 6H). ^{13}C NMR (101 MHz, CDCl_3) δ 154.6, 81.8, 75.4, 25.9. MS (ESI) calculated for $\text{C}_5\text{H}_9\text{O}_3$ [M+H] $^+$: 117.05; found: 116.91. IR (KBr, cm^{-1}) ν = 2986, 2938, 2881, 1797, 1463, 1398, 1288, 1212, 1130, 1058, 988, 776, 709, 602, 553.

4-Phenyl-1,3-dioxolan-2-one (2n):² 79% yield, white solid, mp: 56 – 57 °C, ^1H NMR (400 MHz, CDCl_3) δ 7.48 – 7.33 (m, 5H), 5.67 (t, $J = 8.0$ Hz, 1H), 4.79 (t, $J = 8.4$ Hz, 1H), 4.33 (t, $J = 8.2$ Hz, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 155.0, 135.9, 129.7, 129.2, 126.0, 78.1, 71.2. MS (ESI) calculated for $\text{C}_9\text{H}_9\text{O}_3$ [M+H] $^+$: 165.05; found: 165.00. IR (KBr, cm^{-1}) ν = 2981, 2925, 1795, 1487, 1458, 1393, 1358, 1328, 1169, 1066, 960, 905, 759, 700, 577, 492.

4-(Phenoxymethyl)-1,3-dioxolan-2-one (2o):² 58% yield, white solid, mp: 99 – 100 °C, ^1H NMR (500 MHz, CDCl_3) δ 7.32 – 7.29 (m, 2H), 7.03 – 7.00 (m, 1H), 6.96 – 6.89 (m, 2H), 5.04 – 4.99 (m, 1H), 4.60 (t, $J = 8.4$ Hz, 1H), 4.52 (dd, $J = 8.4, 5.9$ Hz, 1H), 4.23 (dd, $J = 10.6, 4.0$ Hz, 1H), 4.13 (dd, $J = 10.6, 3.6$ Hz, 1H). ^{13}C NMR (126 MHz, CDCl_3) δ 157.8, 154.8, 129.7, 122.0, 114.6, 74.2, 66.9, 66.2. MS (ESI) calculated for $\text{C}_{10}\text{H}_{10}\text{NaO}_4$ [M+Na] $^+$: 217.05; found: 217.05. IR (KBr, cm^{-1}) ν = 2925, 2875, 1801, 1602, 1495, 1455, 1397, 1311, 1251, 1167, 1092, 1057, 1012, 759, 695, 507.

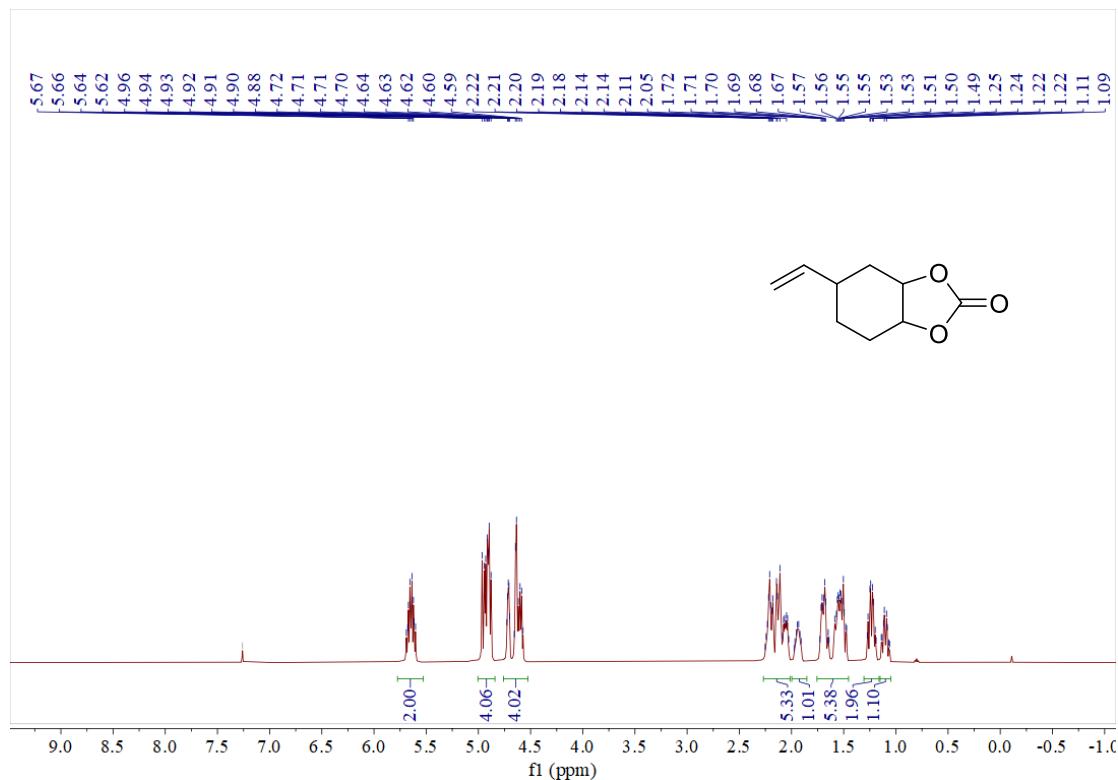
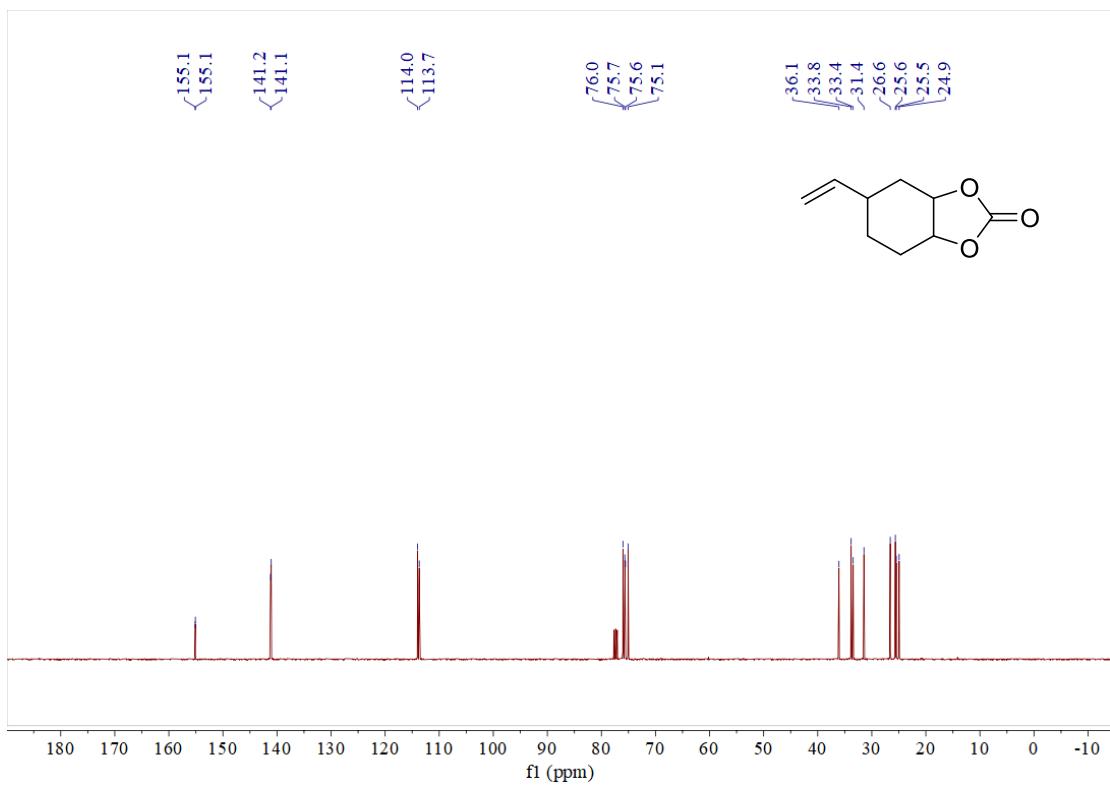
4-((Benzyoxy)methyl)-1,3-dioxolan-2-one (2p):² 84% yield, colorless oil, ^1H NMR (400 MHz, CDCl_3) δ 7.39 – 7.28 (m, 5H), 4.83 – 4.77 (m, 1H), 4.68 – 4.53 (m, 2H), 4.46 (t, $J = 8.4$ Hz, 1H), 4.37 (dd, $J = 8.4, 6.0$ Hz, 1H), 3.71 (dd, $J = 11.0, 3.9$ Hz, 1H), 3.61 (dd, $J = 11.0, 3.7$ Hz, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 155.2, 137.3, 128.6, 128.0, 127.7, 75.3, 73.6, 69.0, 66.3. MS (ESI) calculated for $\text{C}_{11}\text{H}_{12}\text{NaO}_4$ [M+Na] $^+$: 231.06; found: 231.05. IR (KBr, cm^{-1}) ν = 3064, 3031, 2984, 2920, 2868, 1797, 1497, 1478, 1454, 1394, 1360, 1335, 1177, 1105, 1044, 911, 847, 773, 741, 700, 607, 463.

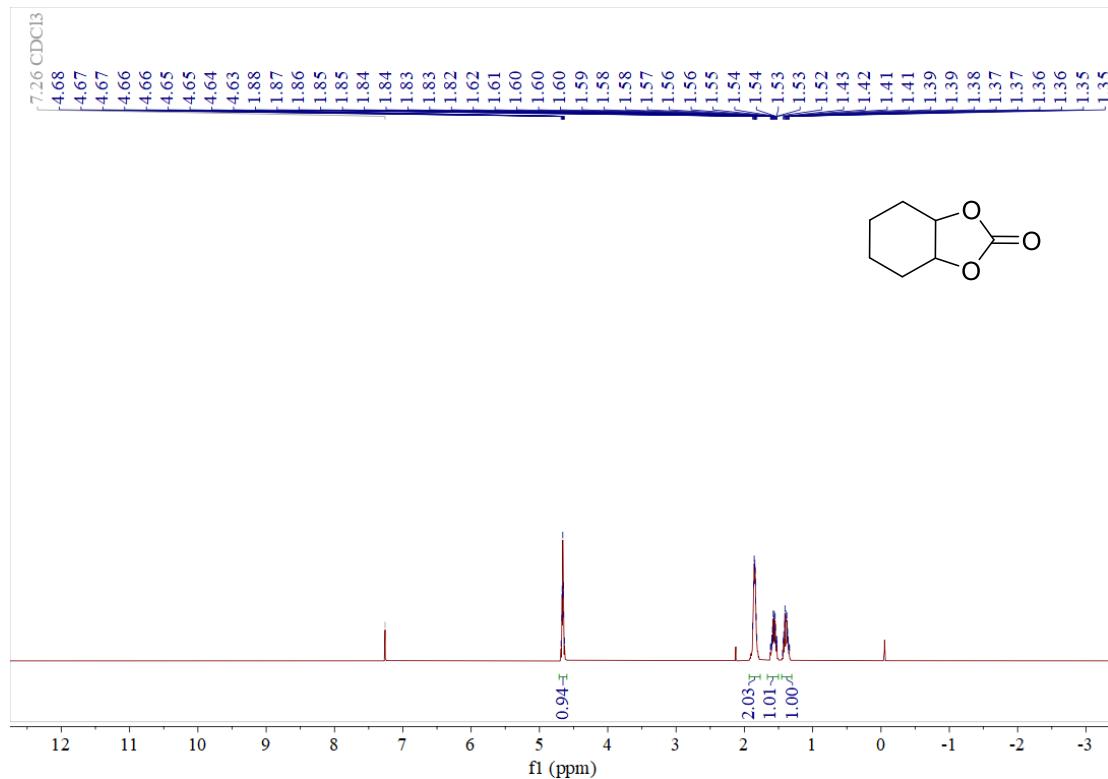
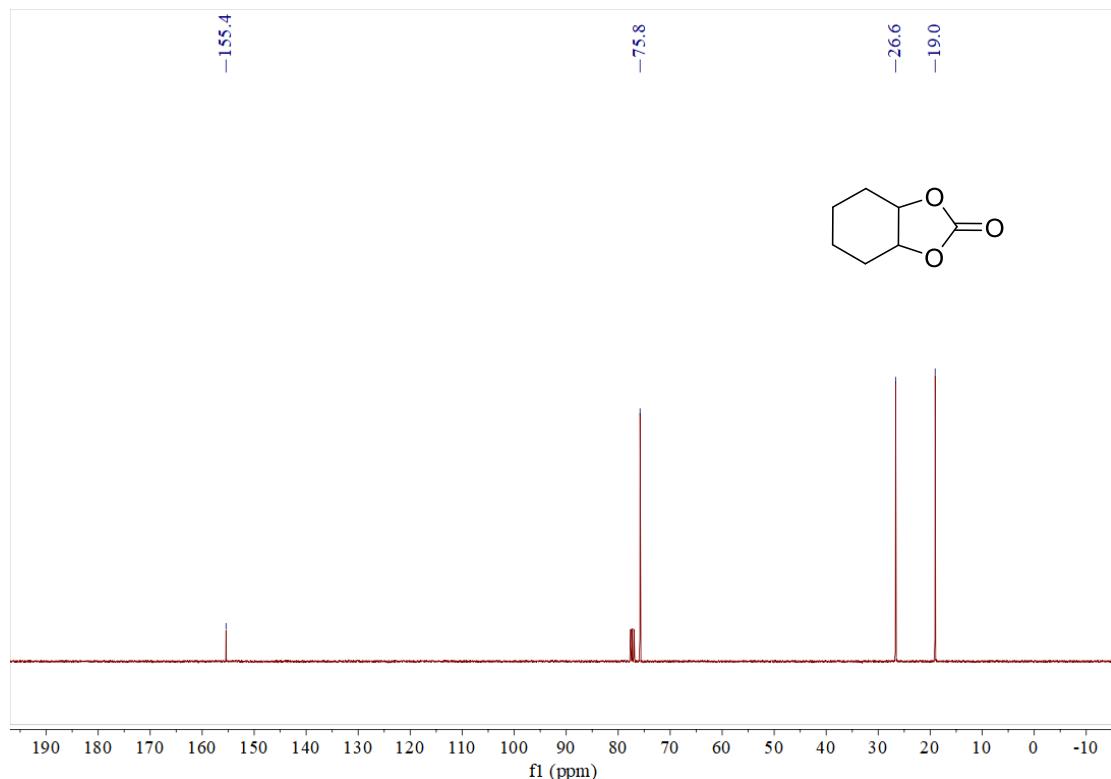
4-((o-Tolyloxy)methyl)-1,3-dioxolan-2-one (2q):⁷ 85% yield, white solid, mp: 95 – 96 °C, ^1H NMR (400 MHz, CDCl_3) δ 7.21 – 7.12 (m, 2H), 6.95 – 6.91 (m, 1H), 6.79 – 6.77 (m, 1H), 5.07 – 5.02 (m, 1H), 4.69 – 4.53 (m, 2H), 4.26 (dd, $J = 10.6, 3.6$ Hz, 1H), 4.13 (dd, $J = 10.6, 3.1$ Hz, 1H), 2.22 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 155.9, 155.0, 131.1, 127.0, 126.9, 121.6, 110.9, 74.4, 67.1, 66.3, 16.0. MS (ESI) calculated for $\text{C}_{11}\text{H}_{12}\text{NaO}_4$ [M+Na] $^+$: 231.06; found: 231.03. IR (KBr, cm^{-1}) ν = 3064, 3018, 2985, 2926, 2879, 1779, 1601, 1499, 1456, 1402, 1293, 1248, 1172, 1126, 1104, 1042, 998, 853, 760, 716, 620, 441.

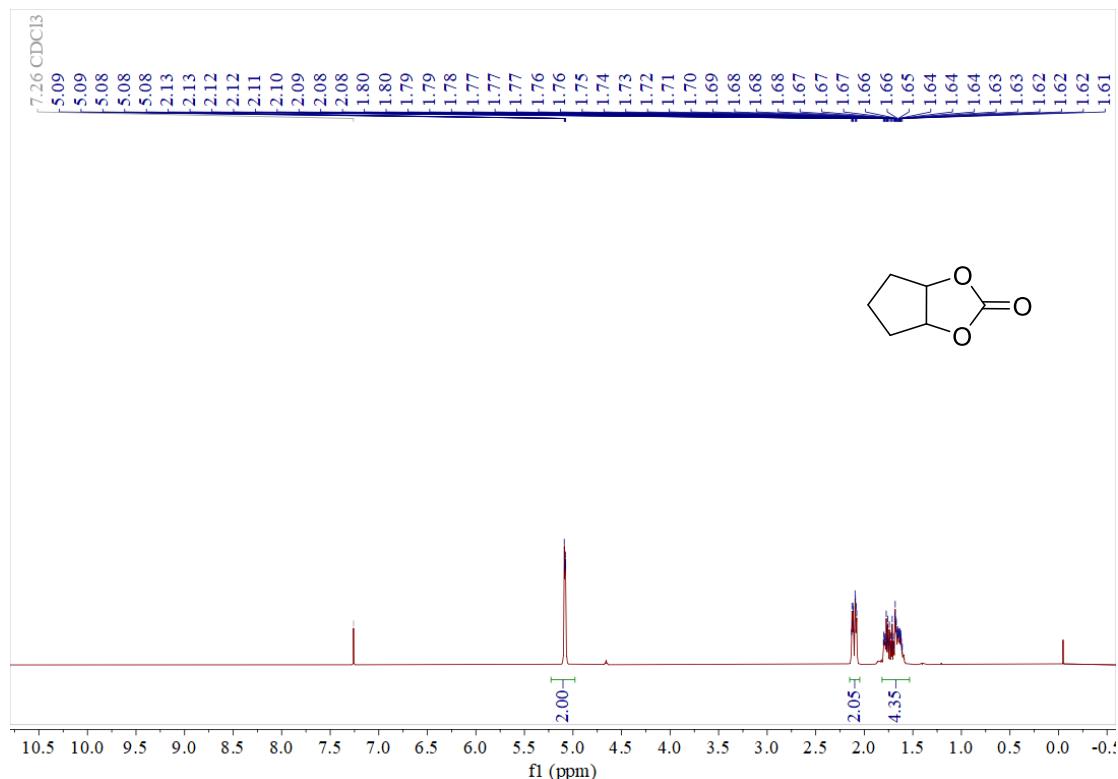
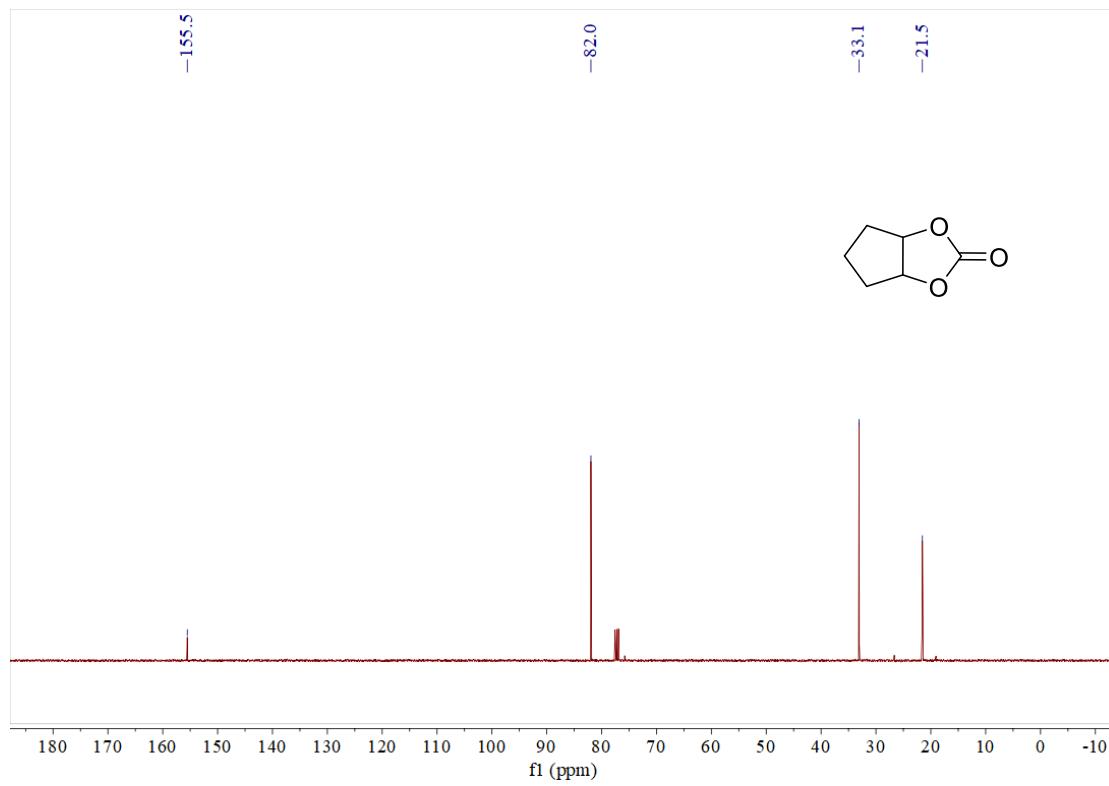
4-((4-Methoxyphenoxy)methyl)-1,3-dioxolan-2-one (2r):⁶ 49% yield, white solid, mp: 99 – 100 °C, ^1H NMR (400 MHz, CDCl_3) δ 6.84 (s, 4H), 5.02 – 4.97 (m, 1H), 4.60 (t, $J = 8.4$ Hz, 1H), 4.52 (dd, $J = 8.4, 5.9$ Hz, 1H), 4.18 (dd, $J = 10.6, 4.2$ Hz, 1H), 4.09 (dd, $J = 10.6, 3.6$ Hz, 1H), 3.77 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 154.7, 152.0, 115.8, 114.8, 74.4, 67.8, 66.2, 55.7. MS (ESI) calculated for $\text{C}_{11}\text{H}_{12}\text{NaO}_5$ [M+Na] $^+$: 247.06; found: 247.03. IR (KBr, cm^{-1}) ν = 2968, 2939, 2846, 1789, 1509, 1446, 1394, 1311, 1236, 1183, 1090, 1027, 869, 825, 777, 754, 717, 522.

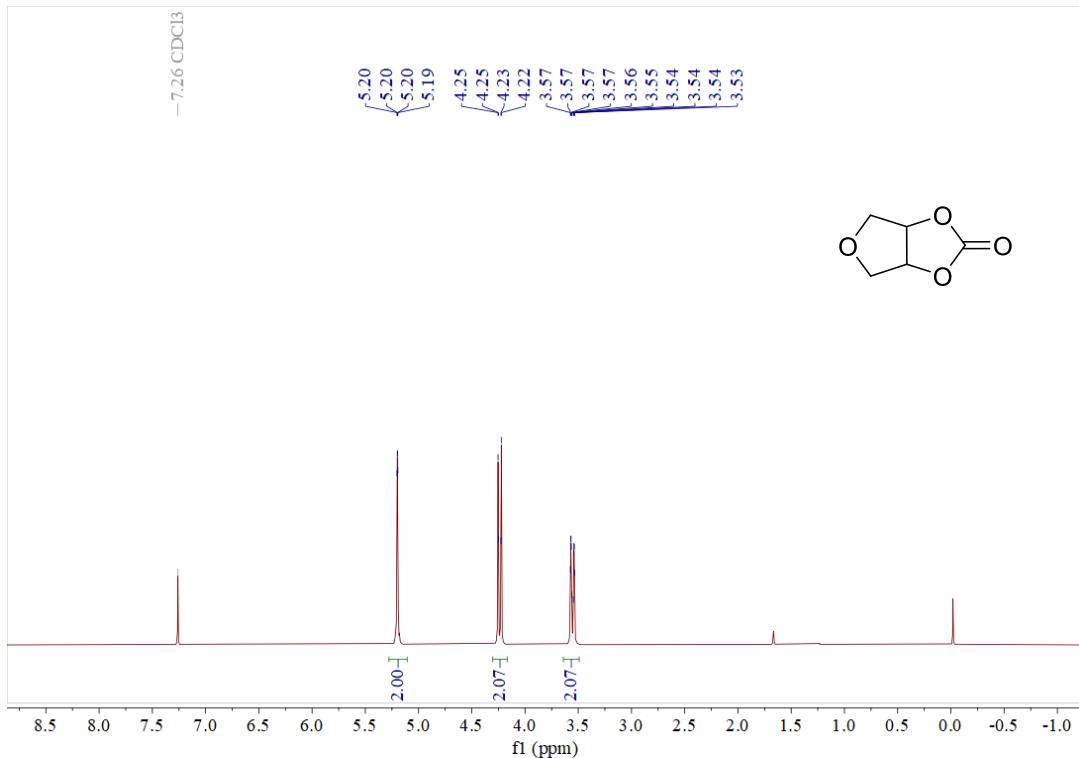
4-((2-Methoxyphenoxy)methyl)-1,3-dioxolan-2-one (2s):⁷ 58% yield, white solid, mp: 72 – 73 °C, ^1H NMR (400

MHz, CDCl₃) δ 7.05 – 6.97 (m, 1H), 6.96 – 6.85 (m, 3H), 5.02 – 4.96 (m, 1H), 4.65 – 4.48 (m, 2H), 4.30 – 4.11 (m, 2H), 3.83 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 154.9, 150.4, 147.5, 123.4, 121.0, 116.6, 112.6, 74.7, 69.3, 66.3, 55.9. MS (ESI) calculated for C₁₁H₁₂NaO₅ [M+Na]⁺: 247.06; found: 247.03. IR (KBr, cm⁻¹) ν = 3080, 3013, 2968, 2917, 2886, 2839, 1782, 1590, 1509, 1453, 1398, 1329, 1257, 1223, 1180, 1153, 1125, 1081, 1050, 1026, 1009, 954, 868, 773, 751, 594, 469.

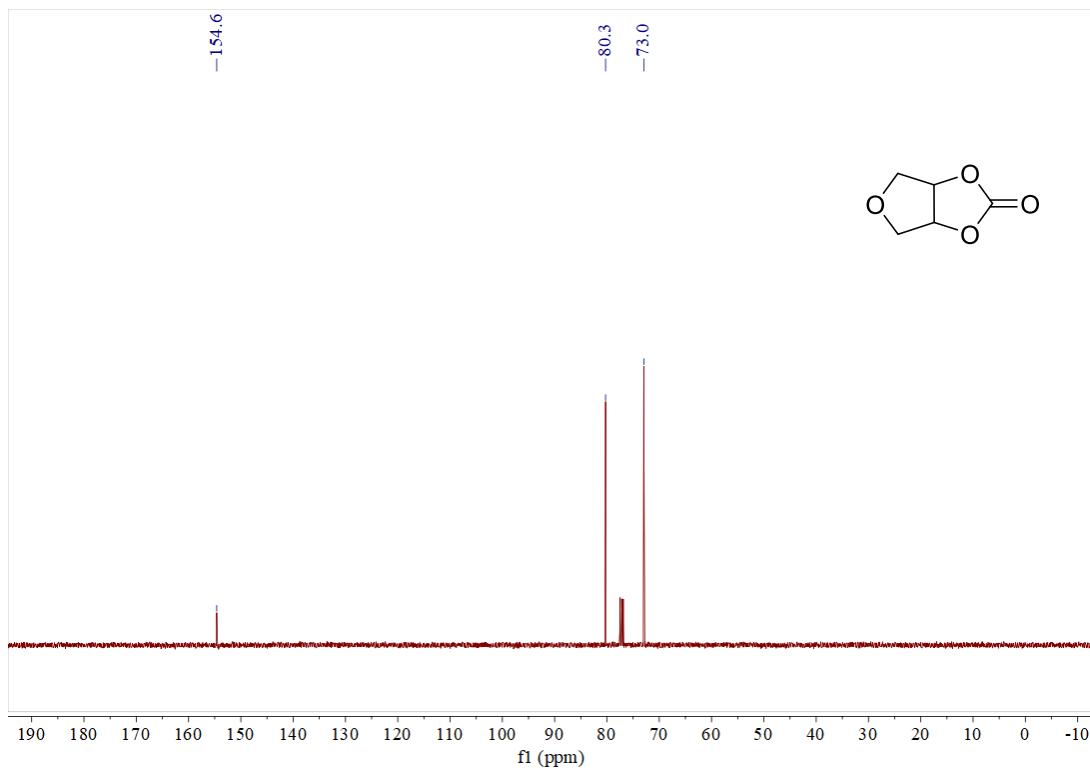
¹H NMR and ¹³C NMR spectra of 2a-2s¹H NMR (500 MHz, CDCl₃) of 5-vinylhexahydrobenzo[d][1,3]dioxol-2-one (2a)¹³C NMR (101 MHz, CDCl₃) of 5-vinylhexahydrobenzo[d][1,3]dioxol-2-one (2a)

¹H NMR (400 MHz, CDCl₃) of hexahydrobenzo[d][1,3]dioxol-2-one (**2b**)¹³C NMR (101 MHz, CDCl₃) of hexahydrobenzo[d][1,3]dioxol-2-one (**2b**)

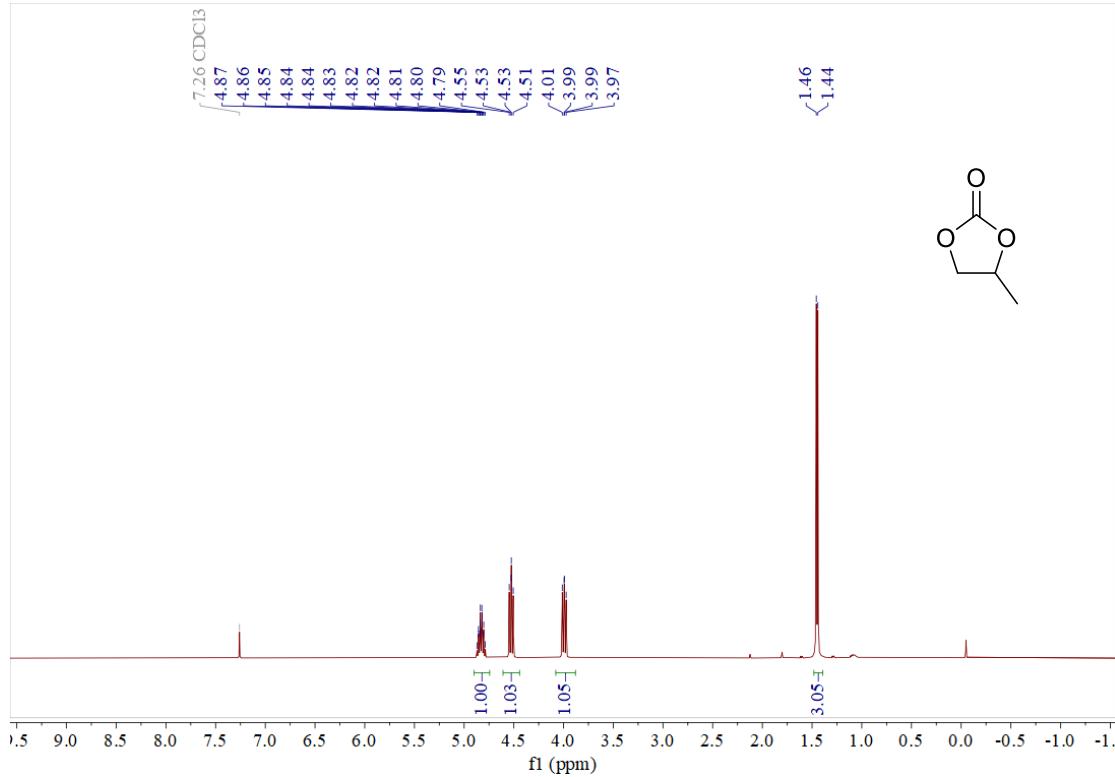
¹H NMR (400 MHz, CDCl₃) of tetrahydro-4H-cyclopenta[d][1,3]dioxol-2-one (**2c**)¹³C NMR (101 MHz, CDCl₃) of tetrahydro-4H-cyclopenta[d][1,3]dioxol-2-one (**2c**)



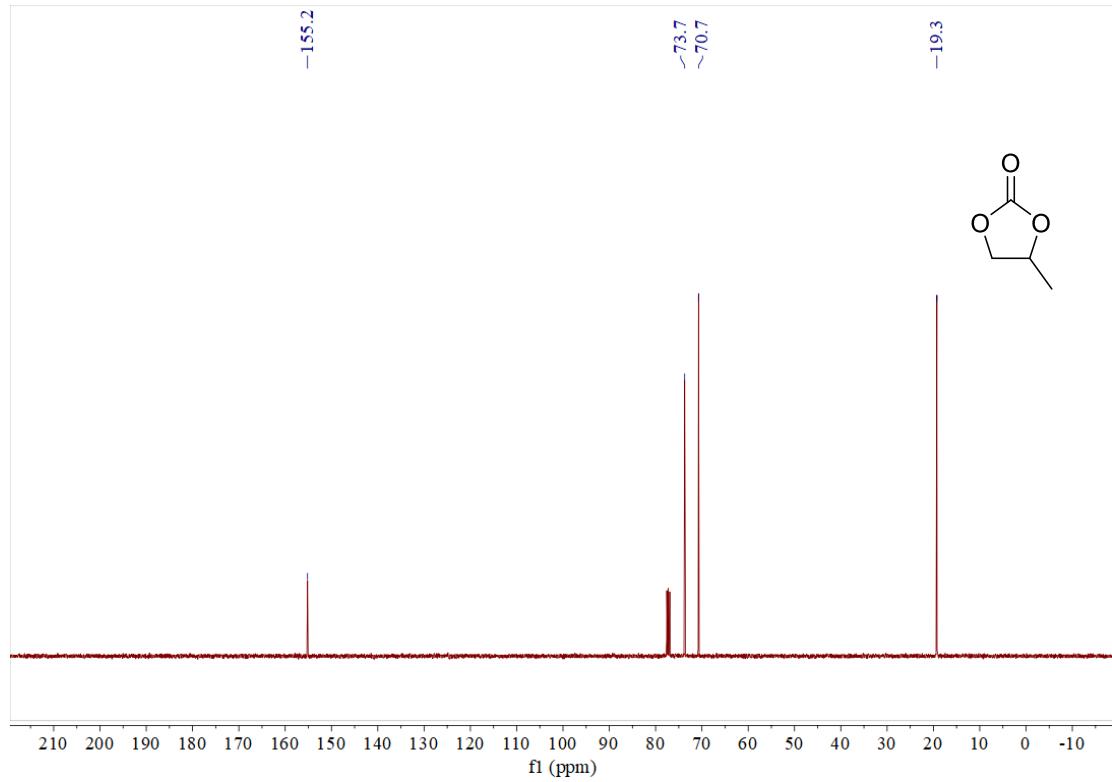
¹H NMR (400 MHz, CDCl₃) of tetrahydrofuro[3,4-d][1,3]dioxol-2-one (**2d**)



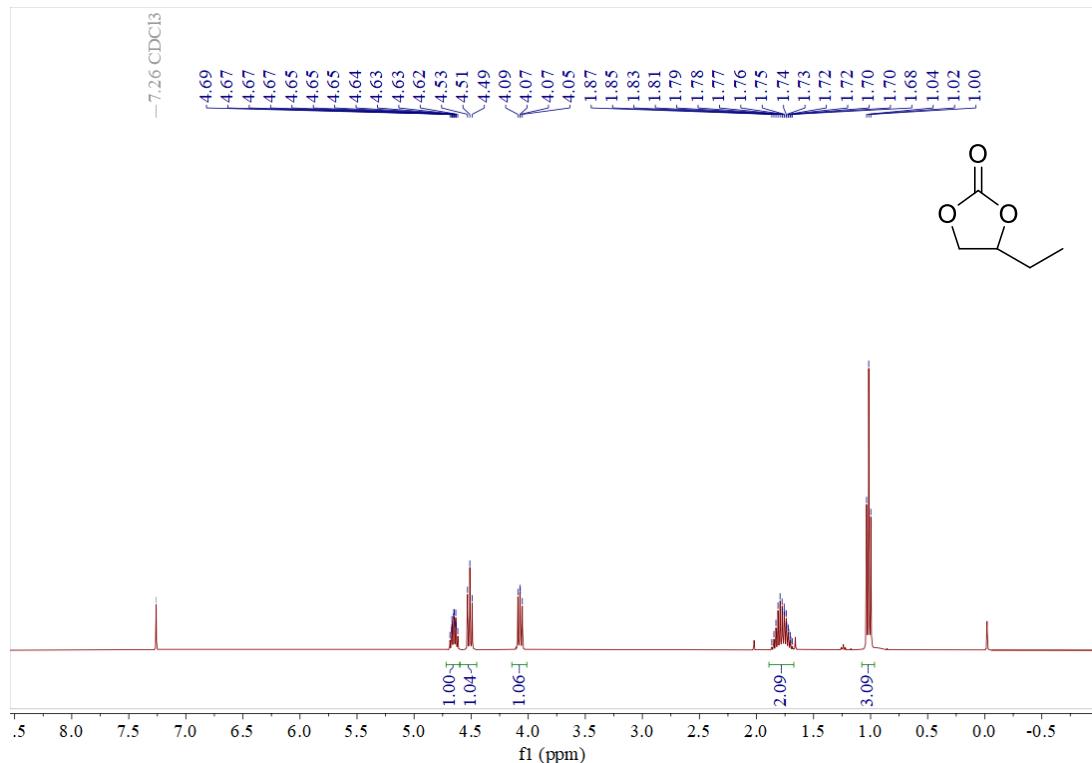
¹³C NMR (101 MHz, CDCl₃) of tetrahydrofuro[3,4-d][1,3]dioxol-2-one (**2d**)



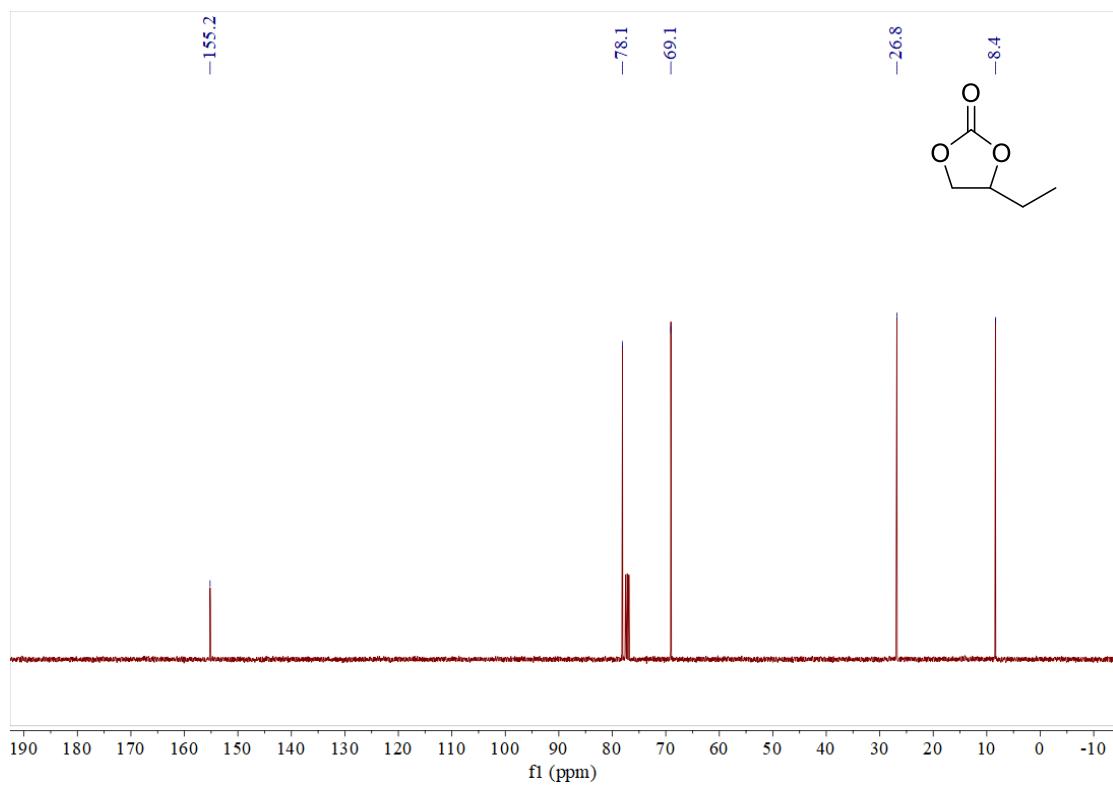
¹H NMR (400 MHz, CDCl₃) of 4-methyl-1,3-dioxolan-2-one (**2e**)



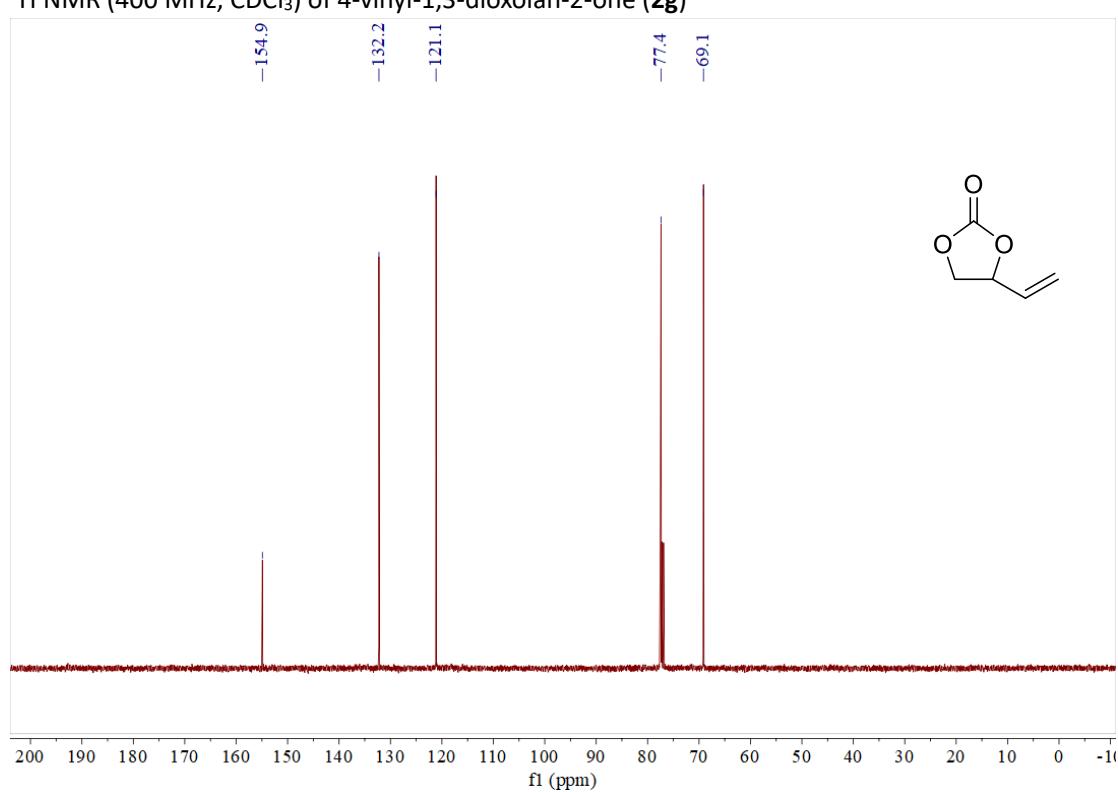
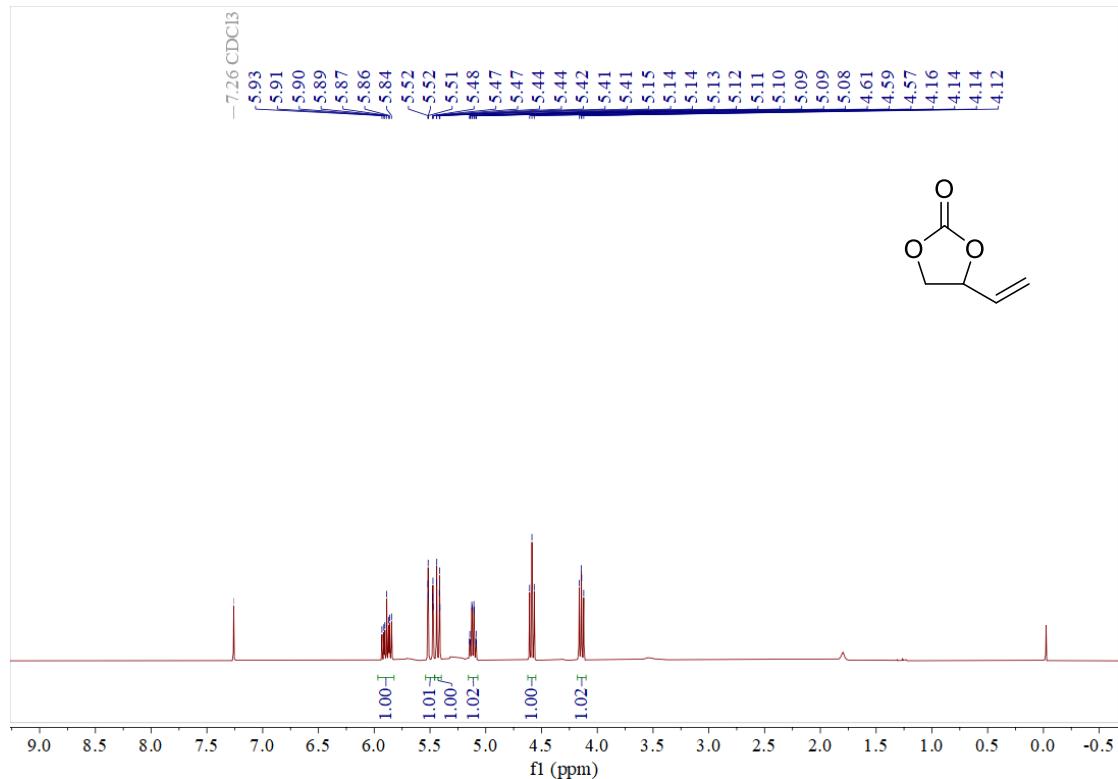
¹³C NMR (101 MHz, CDCl₃) of 4-methyl-1,3-dioxolan-2-one (**2e**)

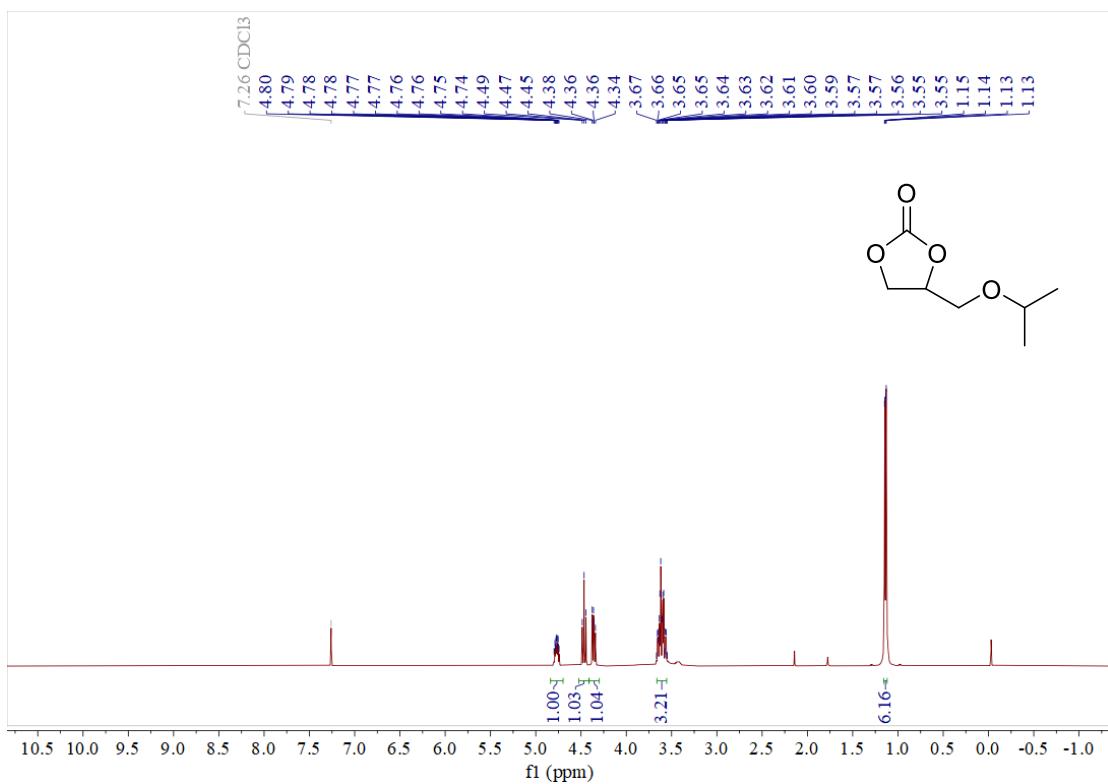


¹H NMR (400 MHz, CDCl₃) of 4-ethyl-1,3-dioxolan-2-one (**2f**)

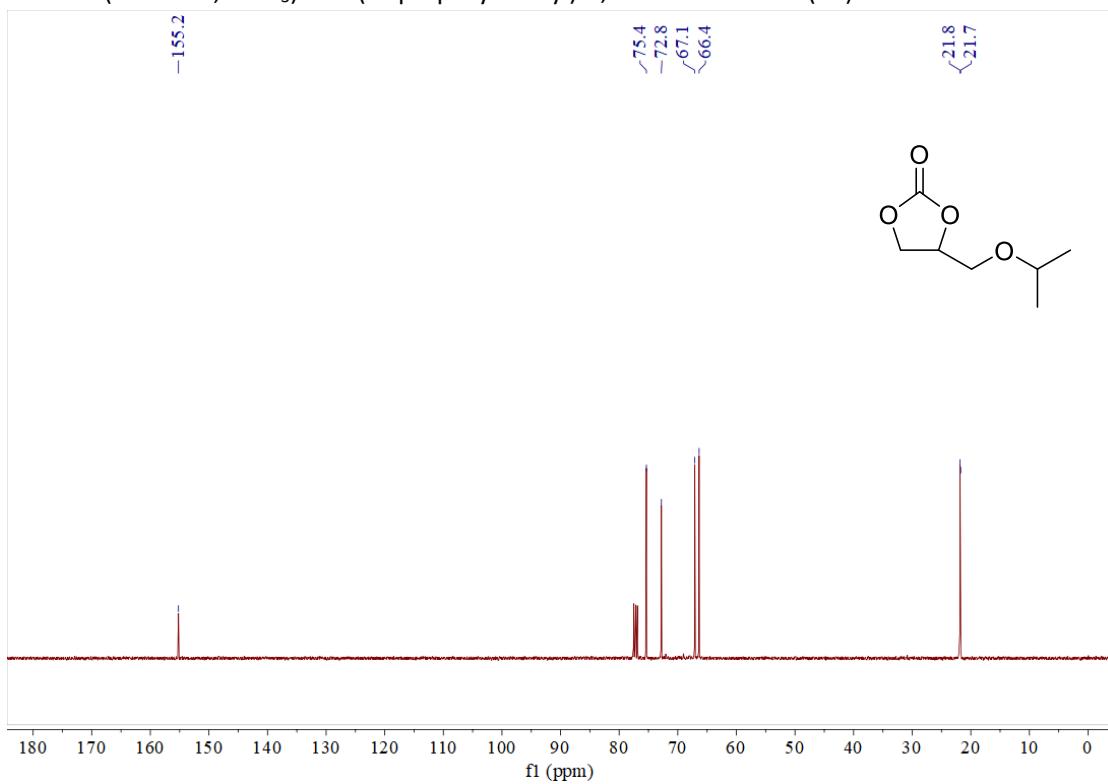


¹³C NMR (101 MHz, CDCl₃) of 4-ethyl-1,3-dioxolan-2-one (**2f**)

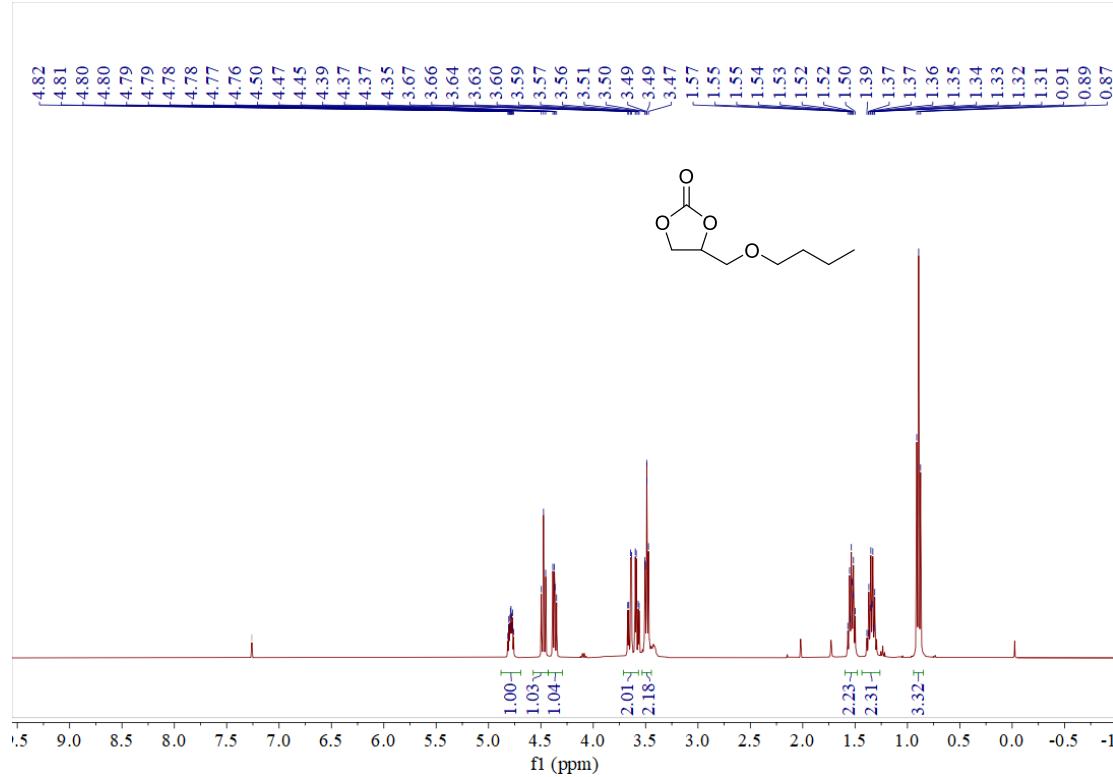
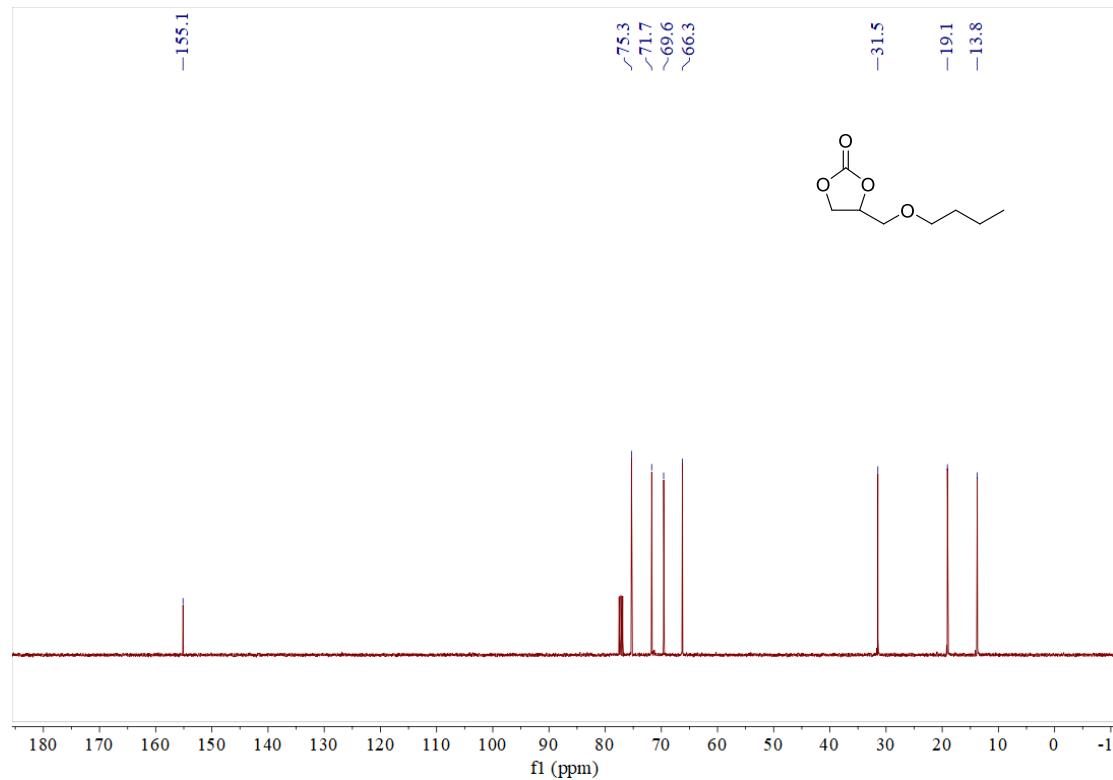


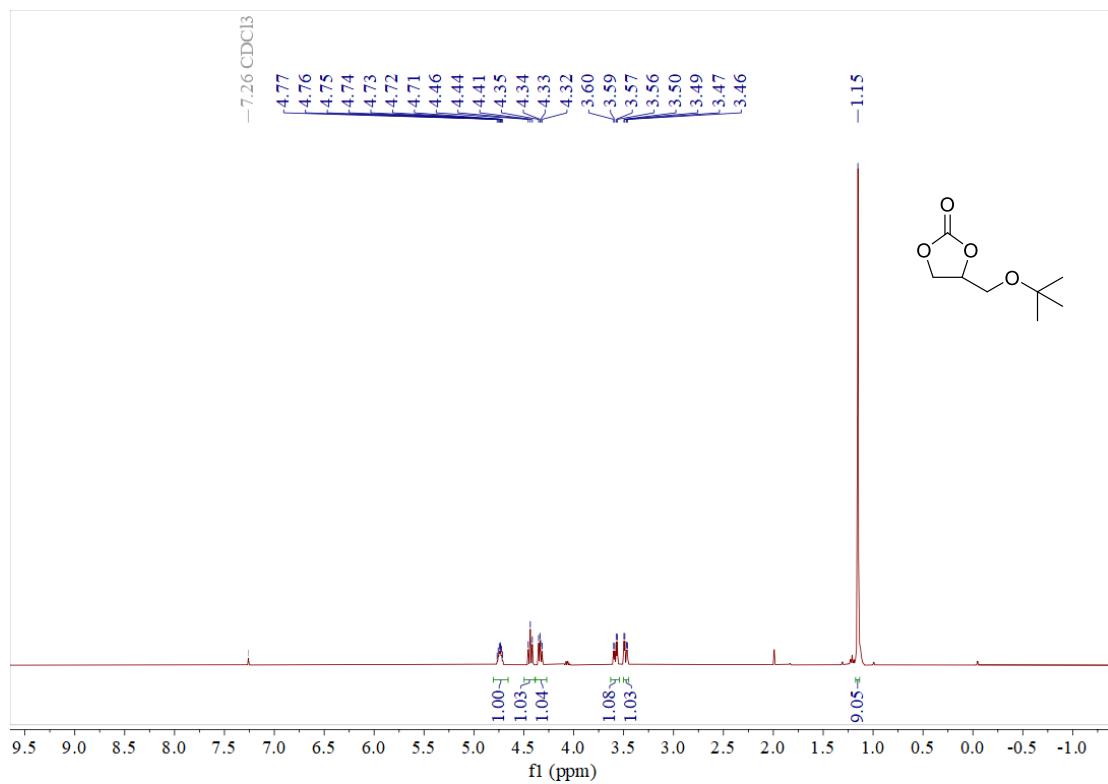
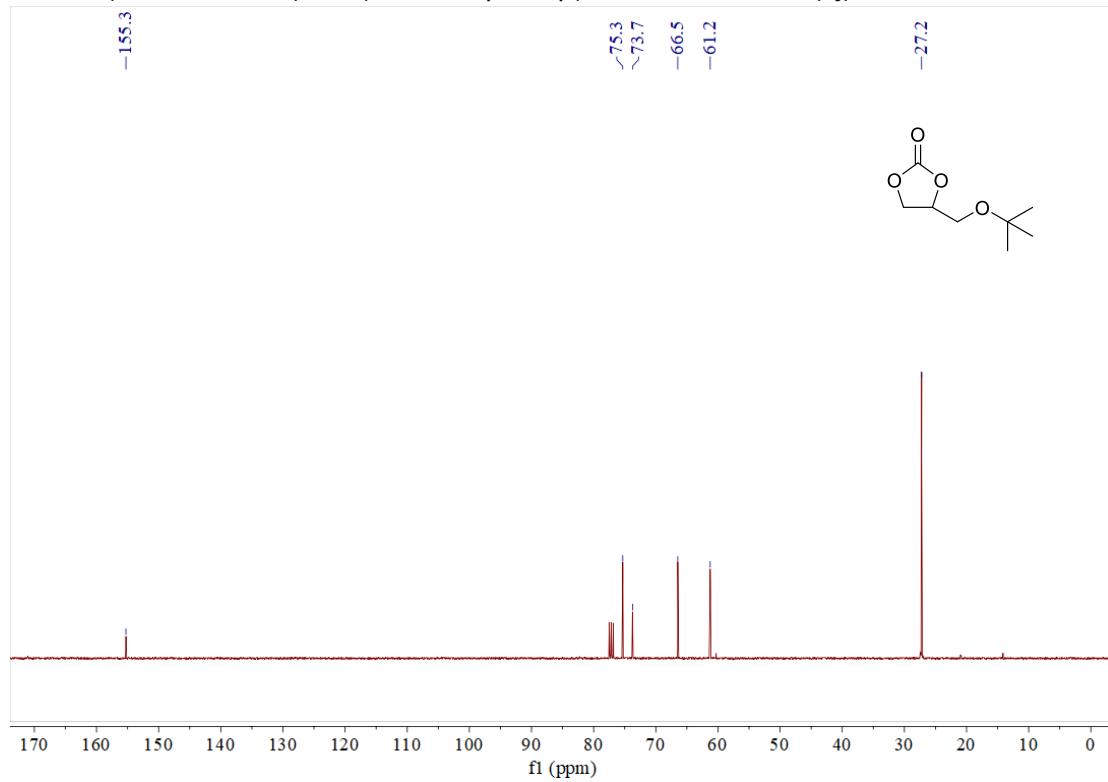


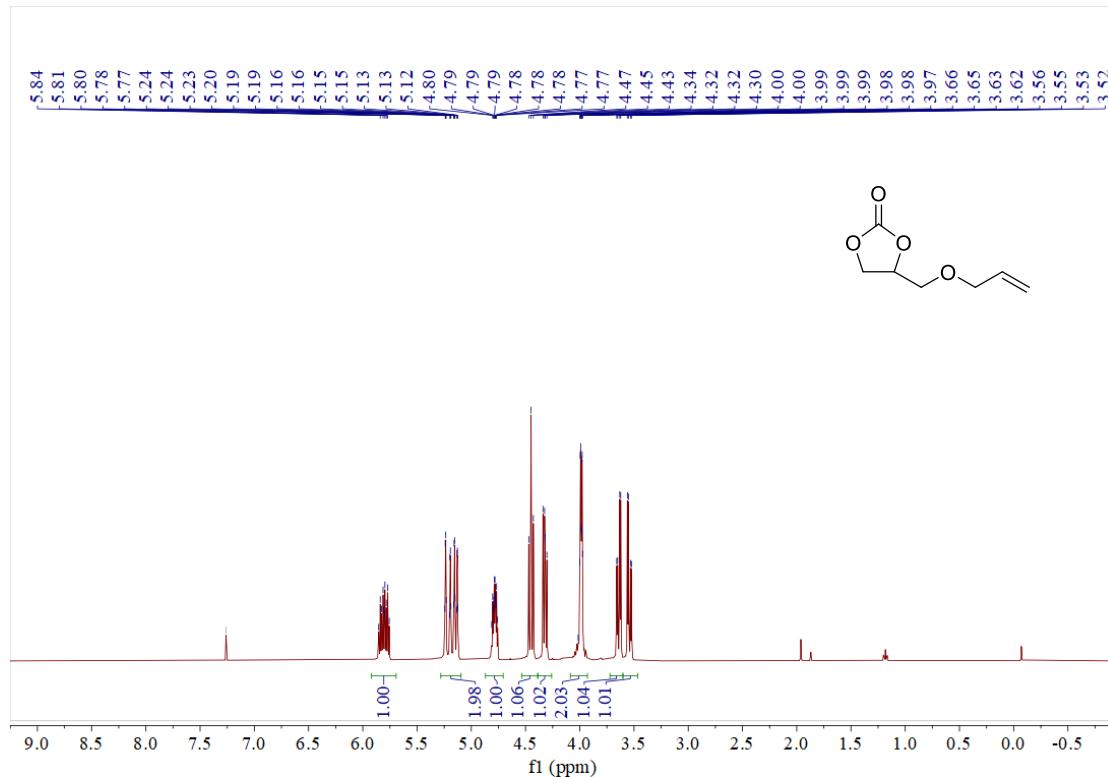
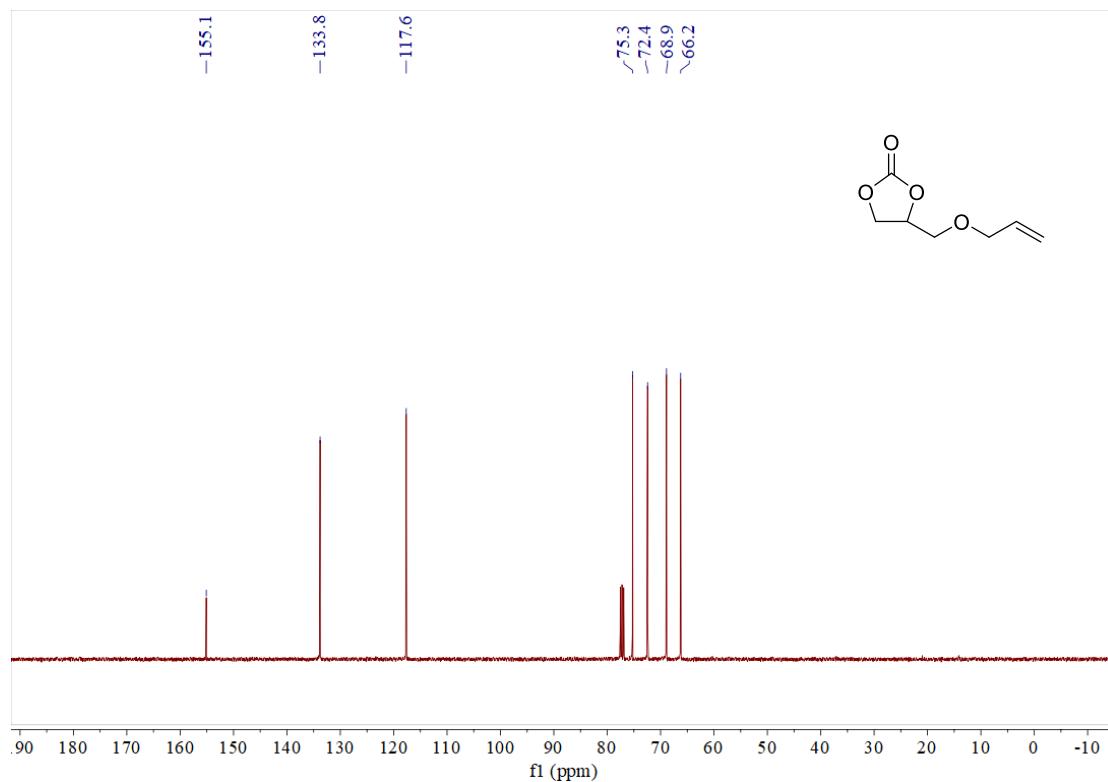
¹H NMR (400 MHz, CDCl₃) of 4-(isopropoxymethyl)-1,3-dioxolan-2-one (**2h**)

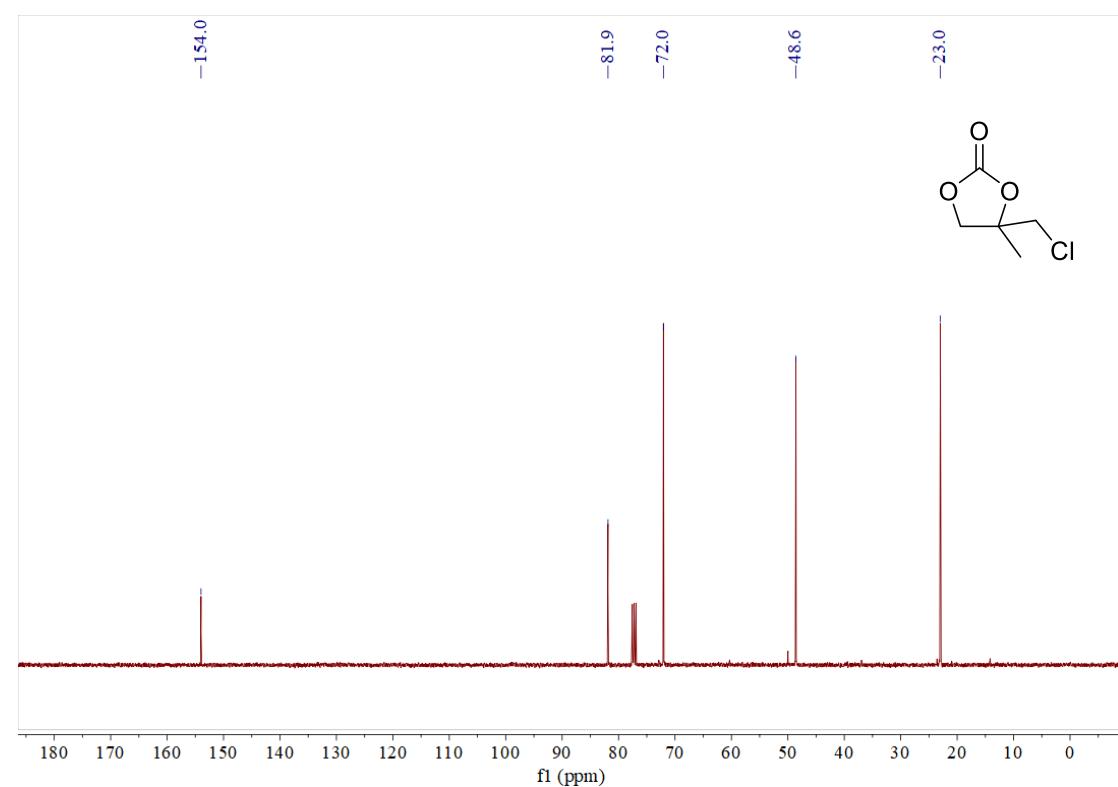
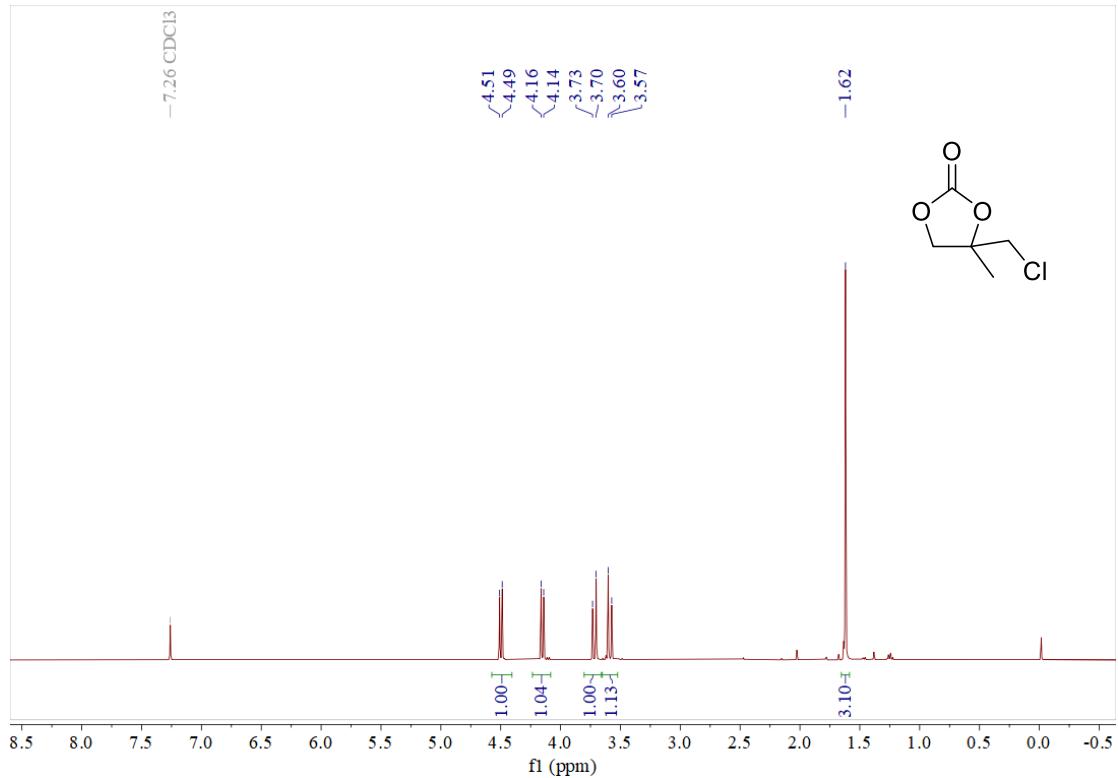


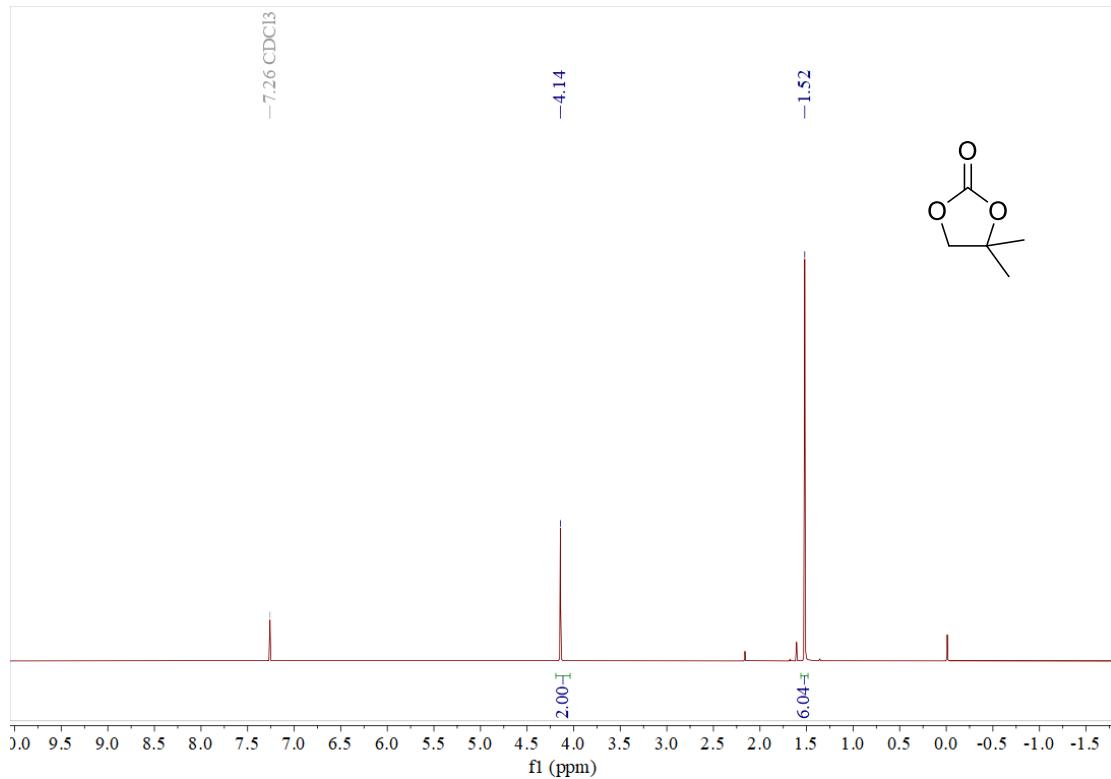
¹³C NMR (101 MHz, CDCl₃) of 4-(isopropoxymethyl)-1,3-dioxolan-2-one (**2h**)

¹H NMR (400 MHz, CDCl₃) of 4-(butoxymethyl)-1,3-dioxolan-2-one (**2i**)¹³C NMR (101 MHz, CDCl₃) of 4-(butoxymethyl)-1,3-dioxolan-2-one (**2i**)

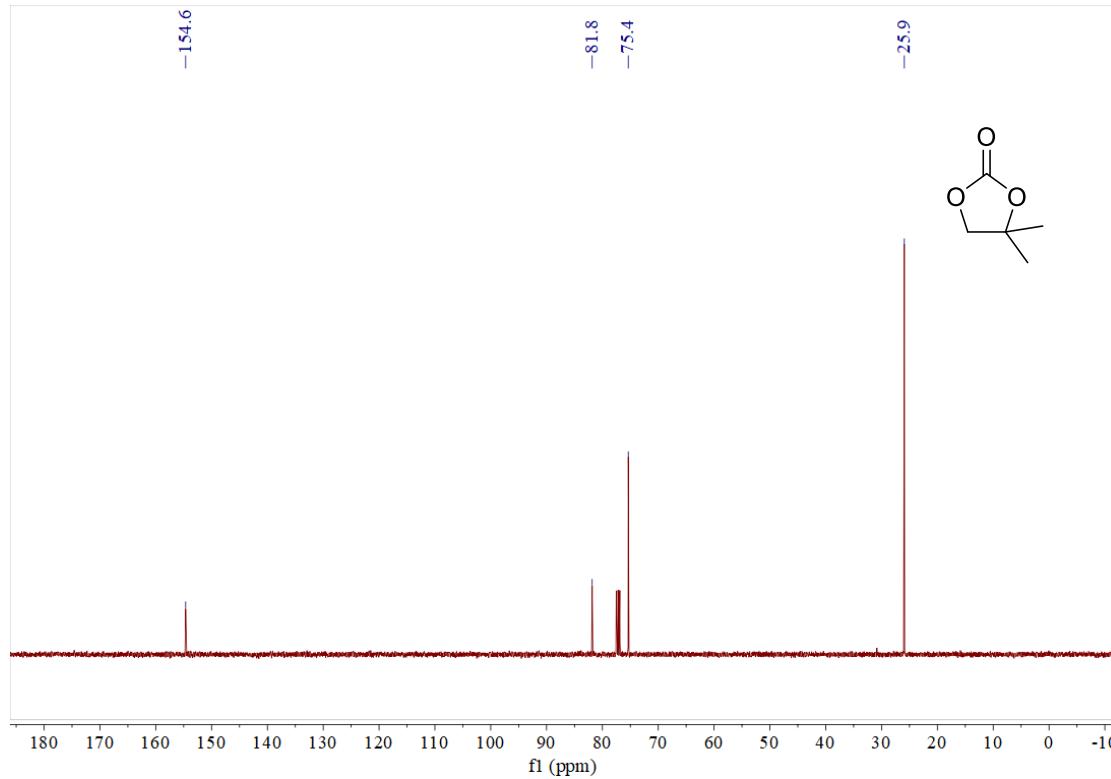
¹H NMR (400 MHz, CDCl₃) of 4-(tert-butoxymethyl)-1,3-dioxolan-2-one (**2j**)¹³C NMR (101 MHz, CDCl₃) of 4-(tert-butoxymethyl)-1,3-dioxolan-2-one (**2j**)

¹H NMR (400 MHz, CDCl₃) of 4-((allyloxy)methyl)-1,3-dioxolan-2-one (**2k**)¹³C NMR (101 MHz, CDCl₃) of 4-((allyloxy)methyl)-1,3-dioxolan-2-one (**2k**)

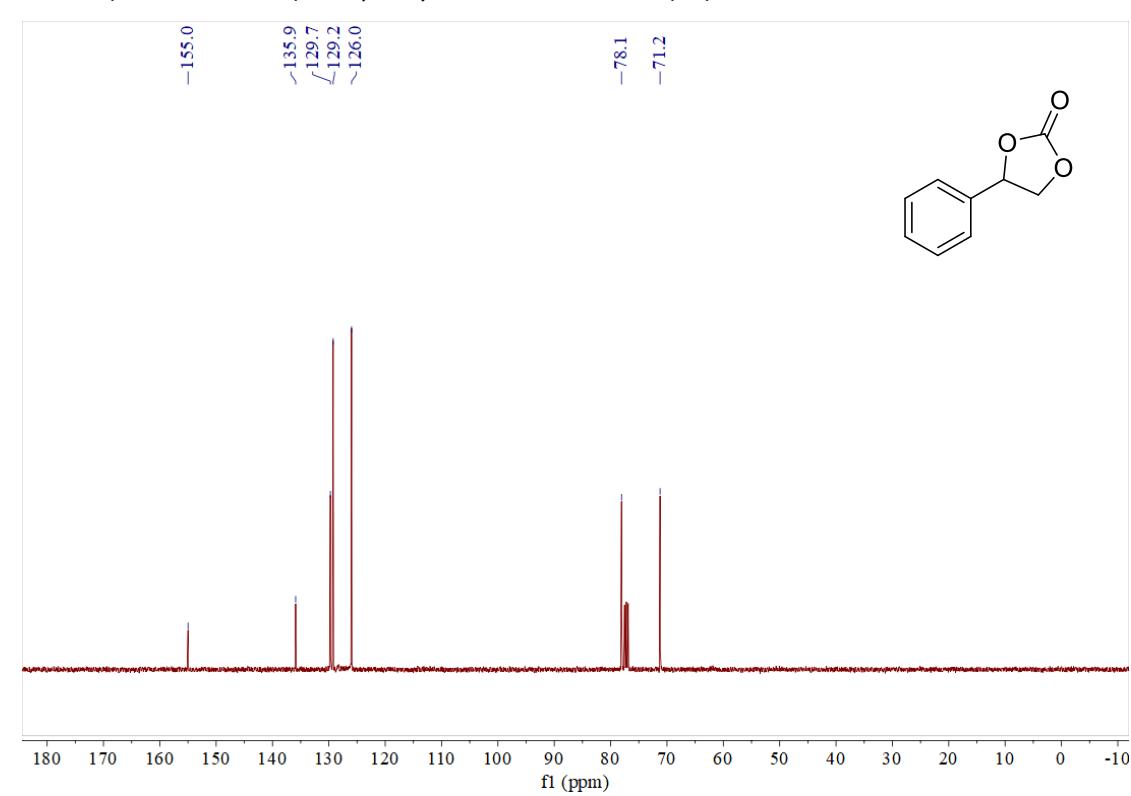
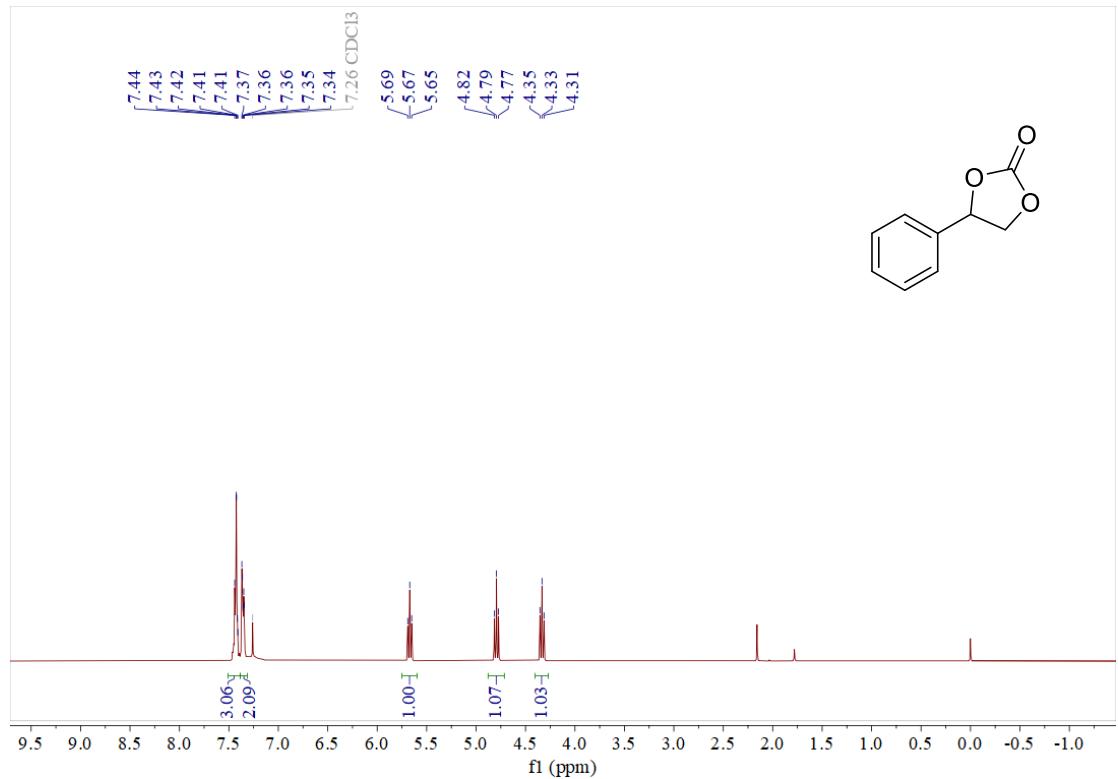


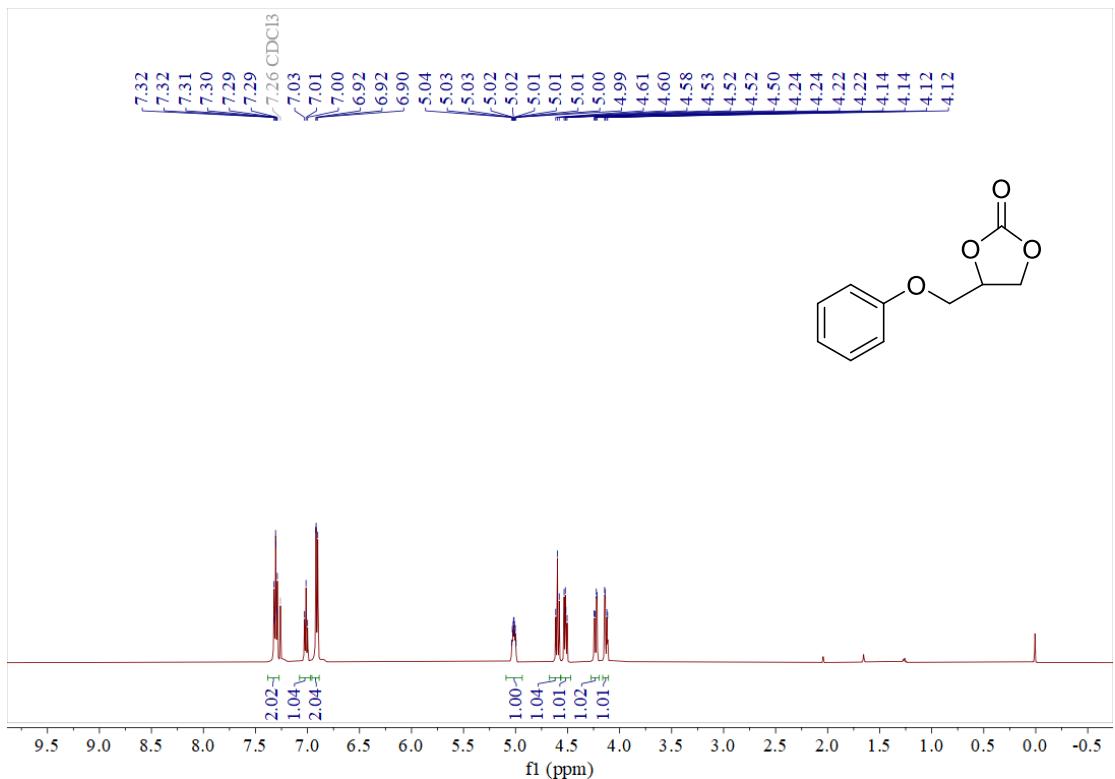


¹H NMR (400 MHz, CDCl₃) of 4,4-dimethyl-1,3-dioxolan-2-one (**2m**)

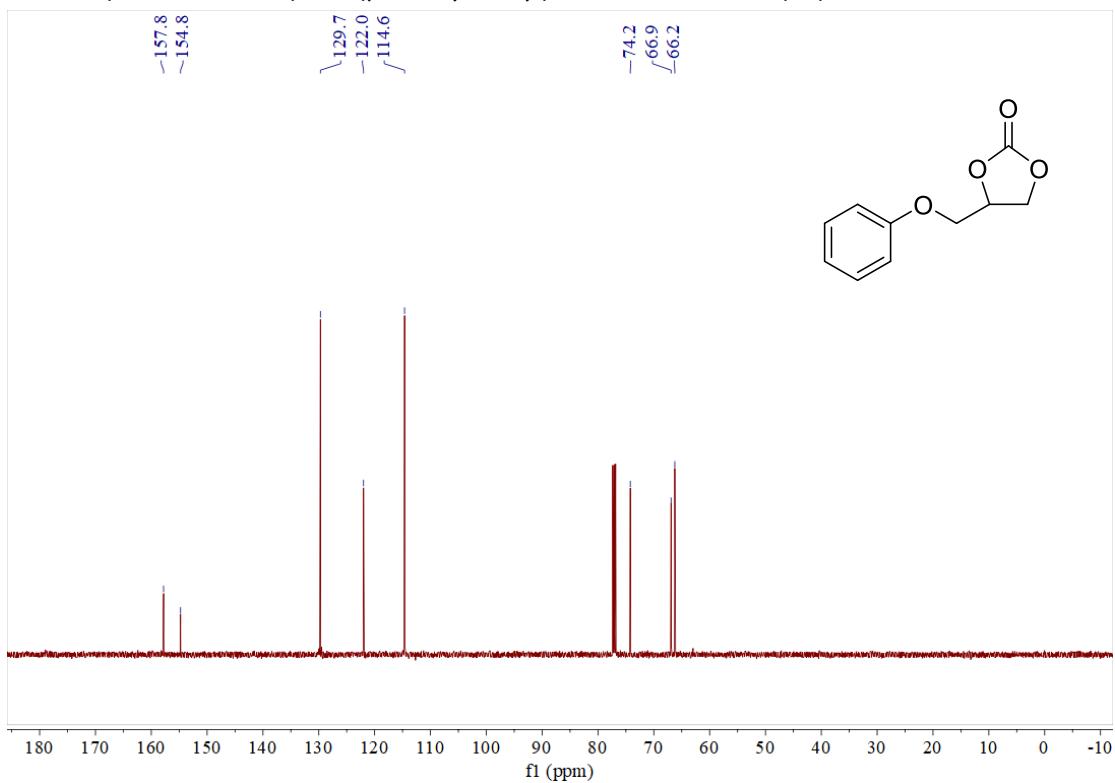


¹³C NMR (101 MHz, CDCl₃) of 4,4-dimethyl-1,3-dioxolan-2-one (**2m**)

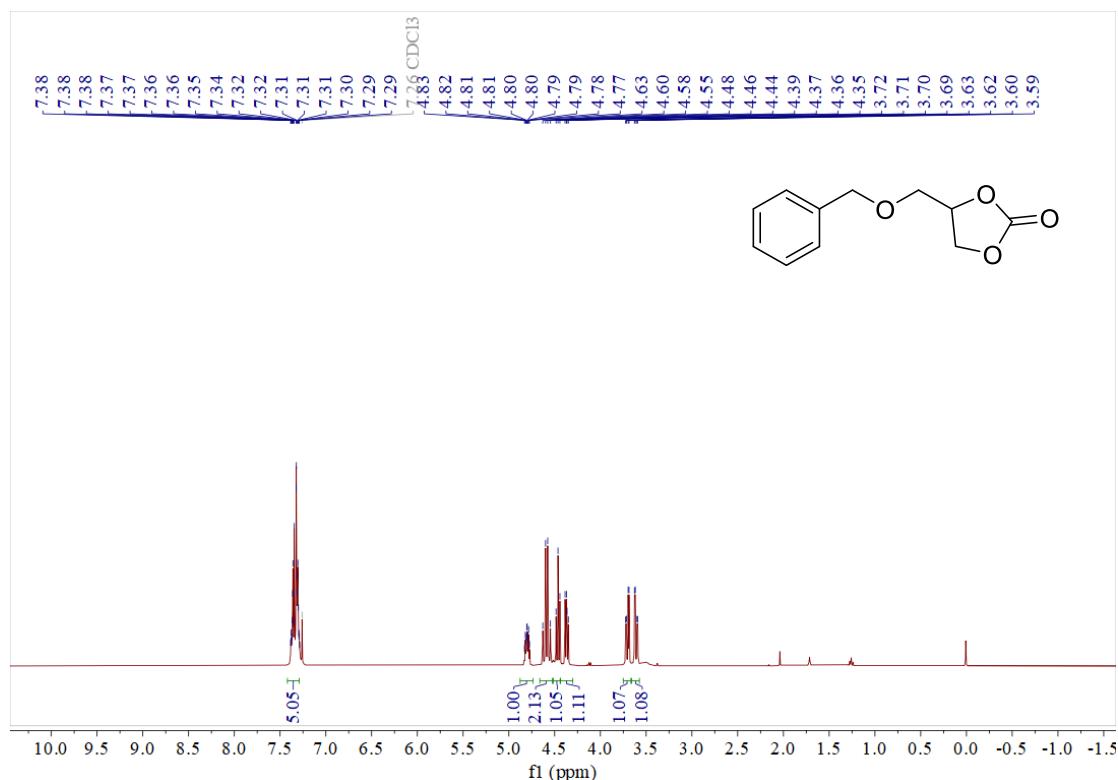




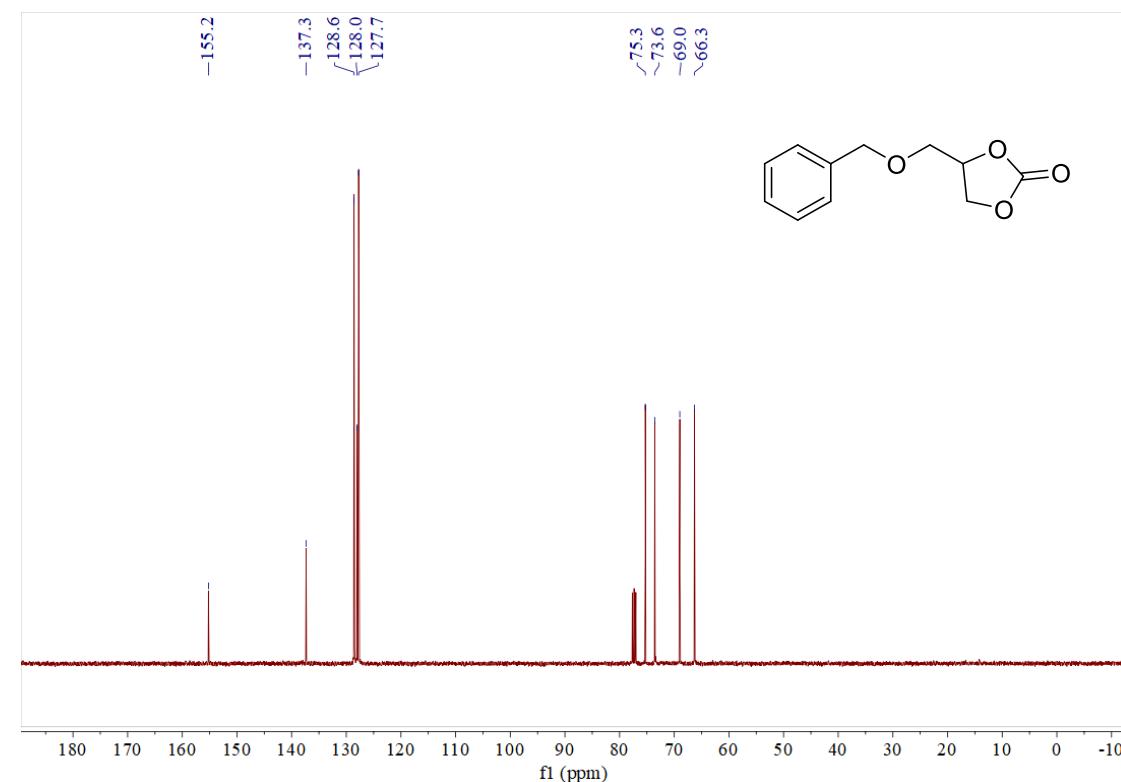
¹H NMR (500 MHz, CDCl₃) of 4-(phenoxy)methyl)-1,3-dioxolan-2-one (**2o**)



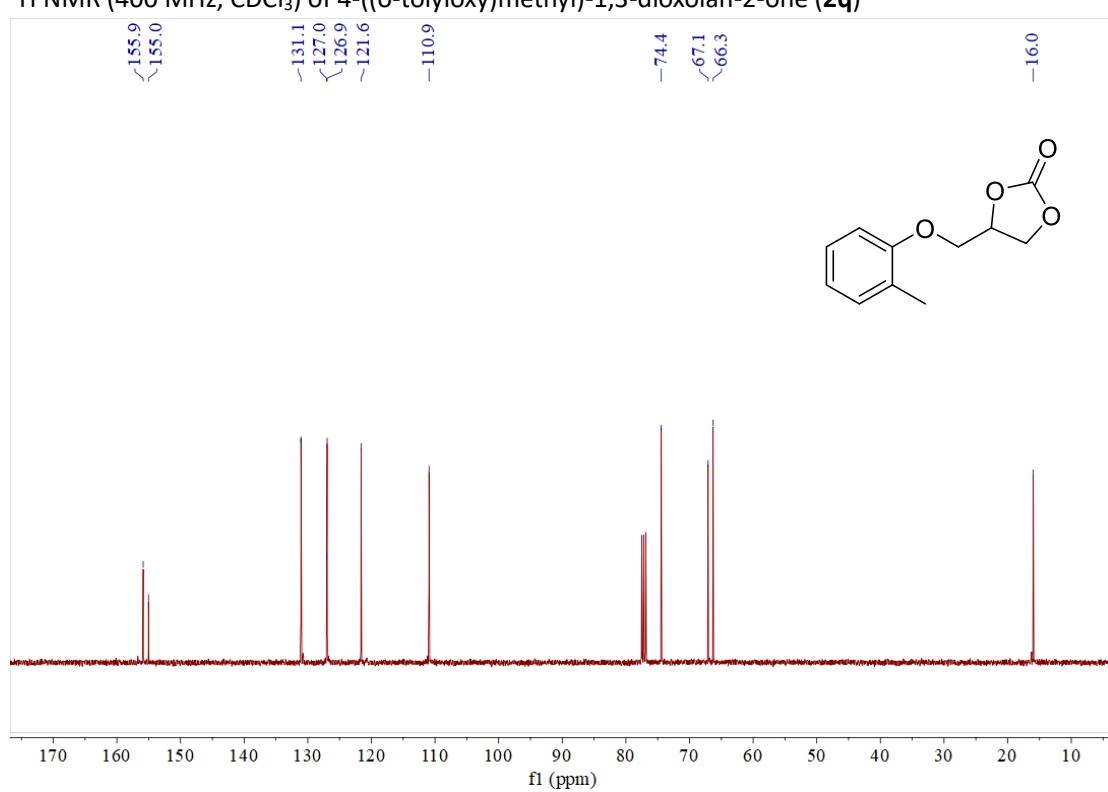
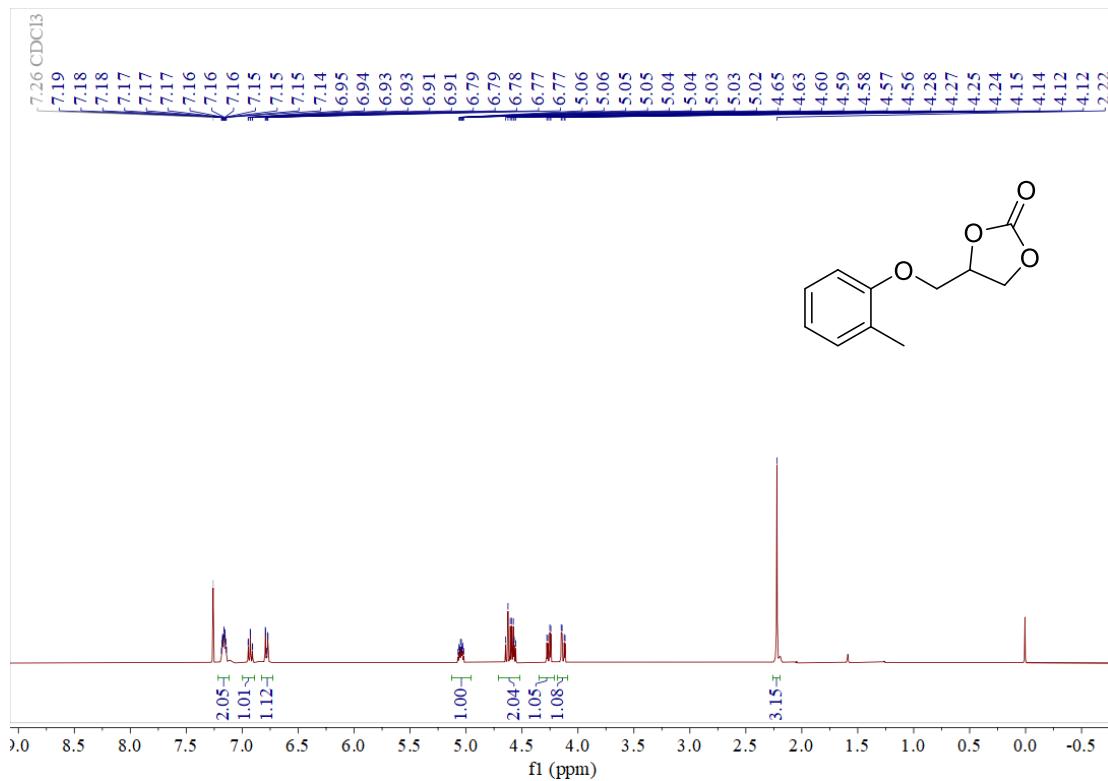
¹³C NMR (126 MHz, CDCl₃) of 4-(phenoxy)methyl)-1,3-dioxolan-2-one (**2o**)

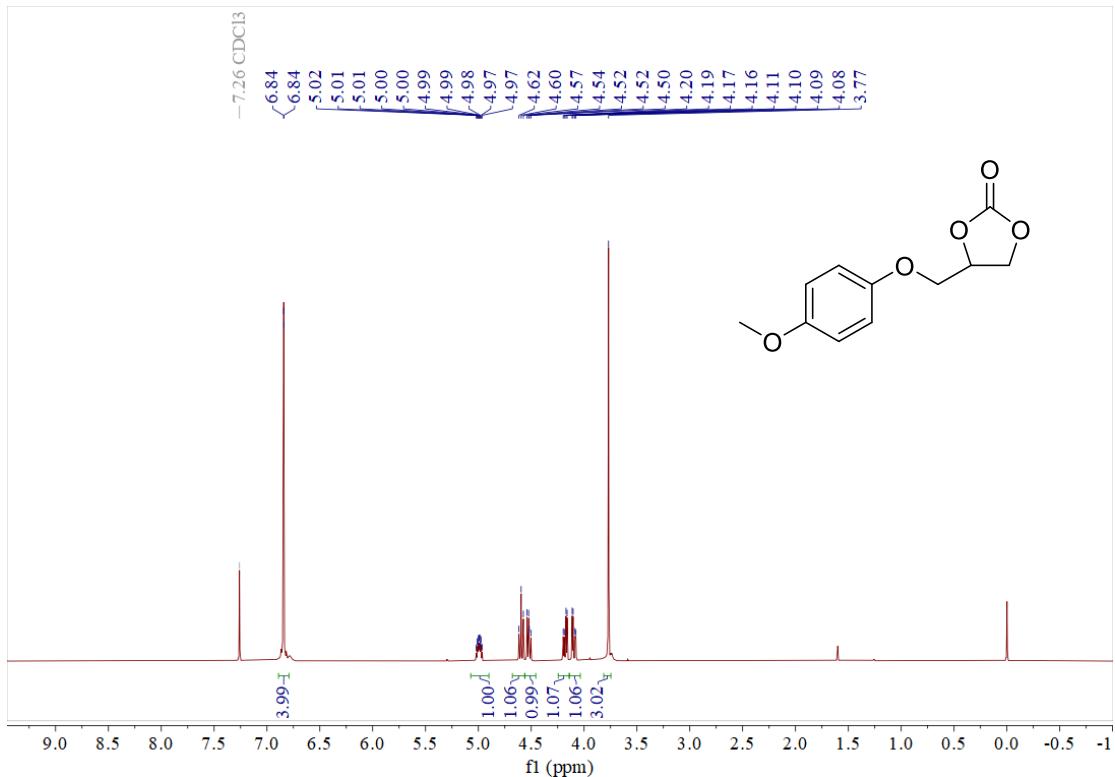
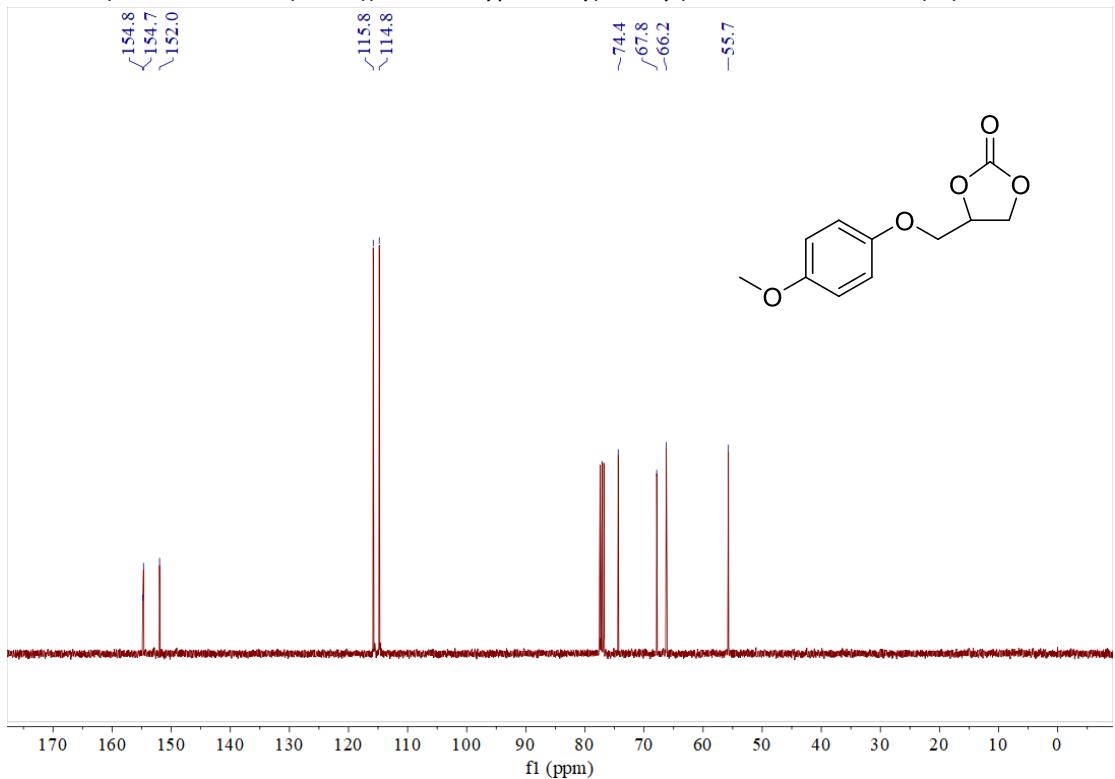


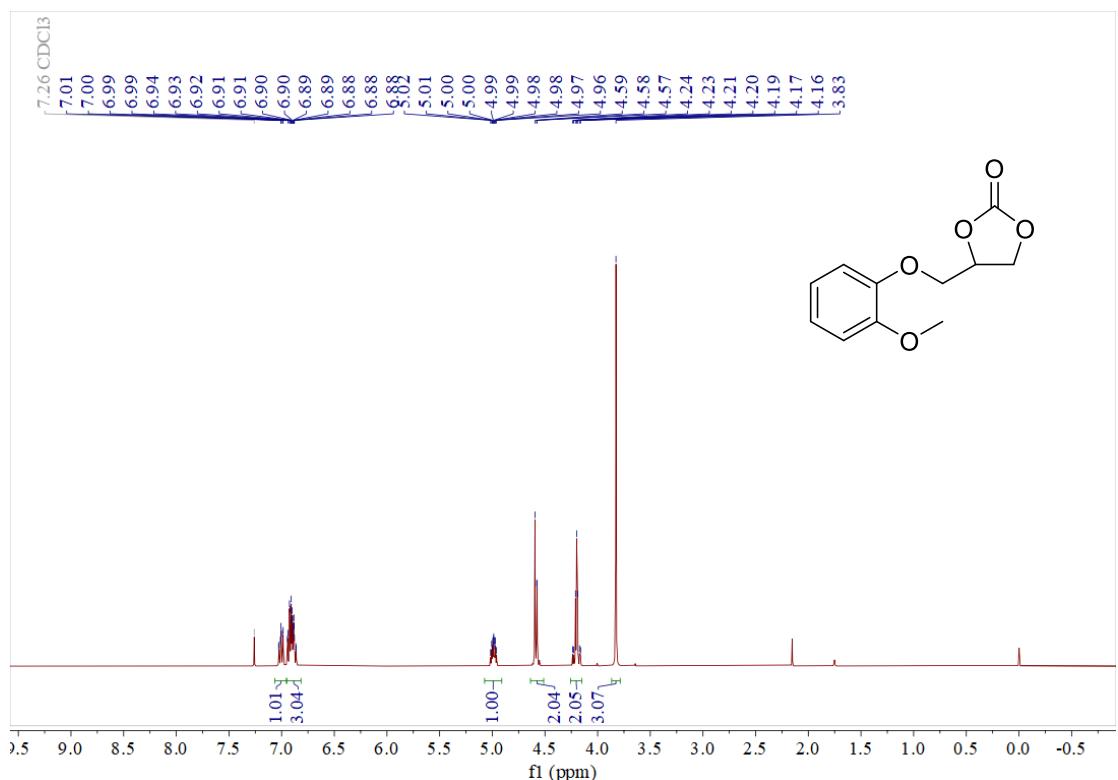
¹H NMR (400 MHz, CDCl₃) of 4-((benzyloxy)methyl)-1,3-dioxolan-2-one (**2p**)



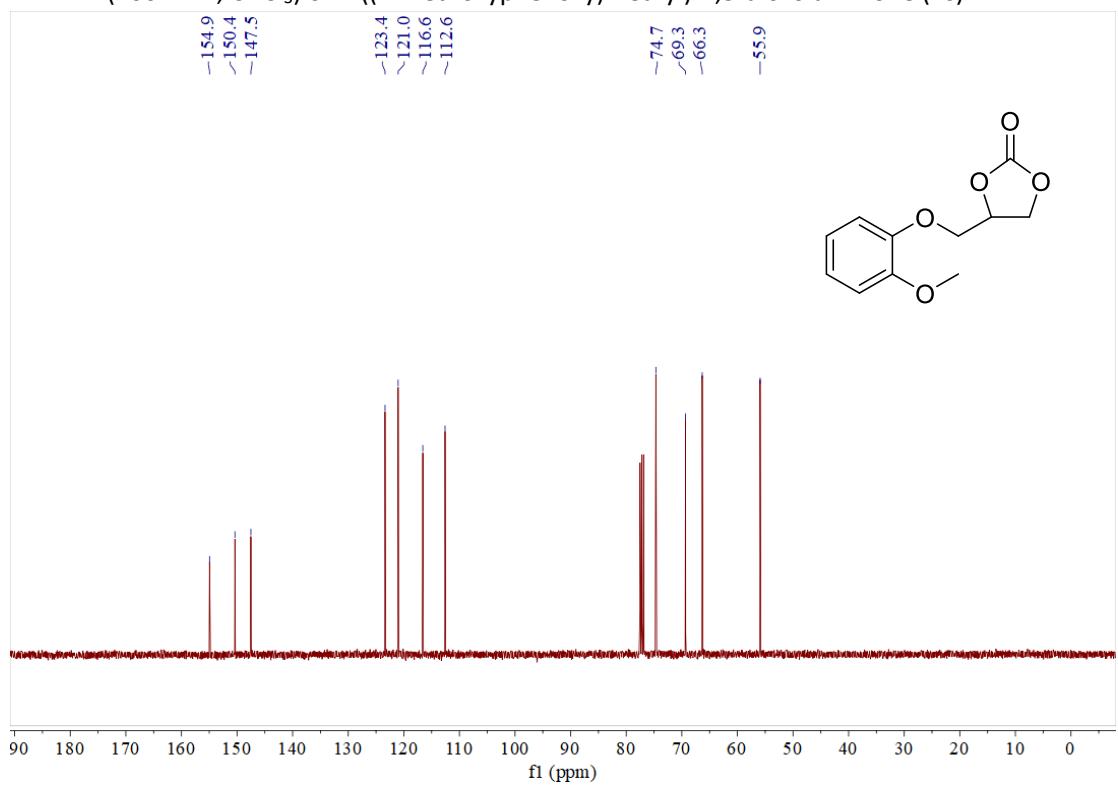
¹³C NMR (101 MHz, CDCl₃) of 4-((benzyloxy)methyl)-1,3-dioxolan-2-one (**2p**)



¹H NMR (400 MHz, CDCl₃) of 4-((4-methoxyphenoxy)methyl)-1,3-dioxolan-2-one (**2r**)¹³C NMR (101 MHz, CDCl₃) of 4-((4-methoxyphenoxy)methyl)-1,3-dioxolan-2-one (**2r**)



¹H NMR (400 MHz, CDCl₃) of 4-((2-methoxyphenoxy)methyl)-1,3-dioxolan-2-one (**2s**)



¹³C NMR (101 MHz, CDCl₃) of 4-((2-methoxyphenoxy)methyl)-1,3-dioxolan-2-one (**2s**)

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