

Supporting information

Boron-Pyridine Nitrogen Cooperative-Catalyzed Conversion of Carbon Dioxide and Epoxides to Cyclic Carbonates

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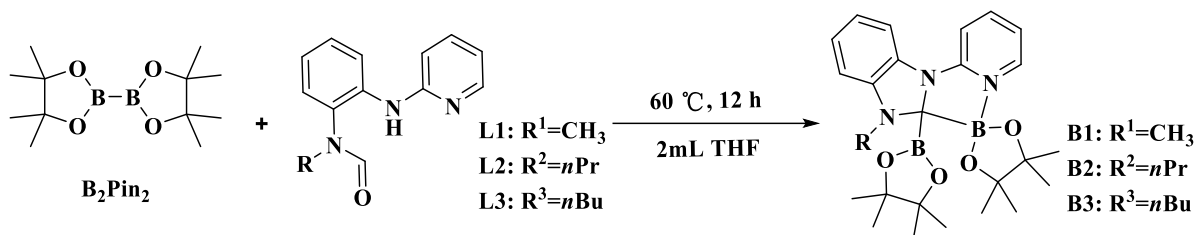
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1. Experimental materials and Instrumentation

General procedure for the synthesis of cyclic carbonates. The epoxides used in this experiment are commercially available and were used as received, unless otherwise indicated. Epoxides (10 mmol), catalyst **B2** (0.1 mmol, 1 mol %) and TBAI (0.4 mmol, 4 mol %) were successively added to the stainless steel reactor with magnetic stirring. The reactor is pressurized to 1.0 MPa with CO₂ and reacted at 50 °C for 21 h. After the reaction, the gas in the reactor is released slowly to atmospheric pressure, and the products were isolated by flash chromatography (petroleum ether: ethyl acetate =5:1-2:1). **Safety reminder:** after 21 h of reaction, take down the reactor for cooling, and slowly release the gas in the reactor to atmospheric pressure after cooling. At this time, do not point the opening releasing gas to the crowd to prevent gas from being sprayed into the eyes.

Analytical Methods. The NMR spectra were recorded on a Bruker Avance III HD 400 spectrometer using TMS as internal standard (400 MHz for ¹H NMR, 100 MHz for ¹³C NMR and 128 MHz for ¹¹B NMR). High-resolution mass spectroscopy (HRMS) data were collected on a UPLC G2-XS QTOF ES+ TOF mass spectrometer. ReactIR spectra were recorded on a METTLER TOLEDO ReactIR 702L.

2. General procedure for the synthesis of B1-B3.¹



General procedure: bis(pinacolato)diboron (**B₂Pin₂**, 0.6 mmol), ring opening ligand **L** (0.5 mmol) and THF (2 ml) were successively added into a 50 mL reaction tube that was equipped with a magnetic stirrer for 12 h at 60 °C. Then the reaction mixture was washed with ethyl acetate, and a yellow solid was collected and dried.

3. Effect of solvent on catalytic activity

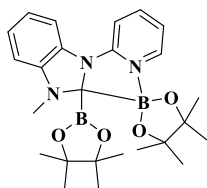
Table S1 Effect of solvent on catalytic activity^a

Entry	T (°C)	Solvent	Yield (%) ^b
1	rt	/	68
2	rt	THF	16
3	rt	MeCN	11
4	rt	toluene	10
5	50	/	64
6	50	THF	58
7	50	MeCN	44
8	50	toluene	52

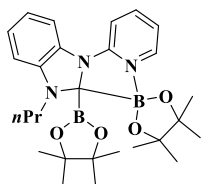
^a Reaction conditions: 1,2-epoxybutane (10.0 mmol), **B2** (0.1 mmol, 1 mol %), TBAI (0.4 mmol, 4 mol %), 1 bar of CO₂, 24 h, neat.

^b Isolated yields were obtained using silica gel column chromatography.

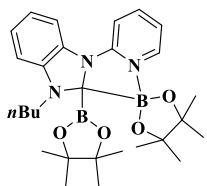
4. Characterization Data for diboron-NHC-adducts B1-B3



diboron-NHC (*N*-heterocyclic carbenes)-adducts B1. give a yellow solid (3.05 g, 66%). ^1H NMR (400 MHz, CDCl_3): δ 8.14-8.12 (m, 1H), 7.59 (t, $J = 8.0$ Hz, 1H), 7.19-7.11 (m, 2H), 6.94 (t, $J = 8.0$ Hz, 1H), 6.85-6.82 (m, 1H), 6.55 (t, $J = 7.6$ Hz, 1H), 6.32 (dd, $J = 8.0$ Hz, $J = 4.0$ Hz, 1H), 3.03 (s, 3H), 1.24 (d, $J = 8.0$ Hz, 24H), ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3): δ 160.4, 149.9, 141.8, 139.5, 136.0, 126.2, 117.5, 115.5, 115.2, 110.3, 106.0, 81.8, 32.3, 26.1, 25.9, 25.9, 24.6, ppm; ^{11}B NMR (128 MHz, CDCl_3): δ 21.98 ppm; HRMS (ESI) calcd for $\text{C}_{25}\text{H}_{36}\text{B}_2\text{N}_3\text{O}_4$ $[\text{M}+\text{H}]^+$ 464.2892, found 464.2895.



diboron-NHC (*N*-heterocyclic carbenes)-adducts B2.¹ give a yellow solid (3.01 g, 62%). ^1H NMR (400 MHz, CDCl_3): δ 8.10 (d, $J = 4.0$ Hz, 1H), 7.56 (t, $J = 8.0$ Hz, 1H), 7.13 (dd, $J = 16.0$ Hz, $J = 8.0$ Hz, 2H), 6.90 (t, $J = 7.6$ Hz, 1H), 6.79 (t, $J = 6.4$ Hz, 1H), 6.50 (t, $J = 7.6$ Hz, 1H), 6.31 (d, $J = 8.0$ Hz, 1H), 3.27 (t, $J = 8.0$ Hz, 2H), 1.70-1.60 (m, 2H), 1.22 (d, $J = 8.0$ Hz, 24H), 0.90 (t, $J = 8.0$ Hz, 3H), ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3): δ 159.8, 149.4, 141.8, 139.4, 135.6, 126.1, 117.4, 115.3, 114.5, 110.1, 105.3, 81.7, 48.8, 26.1, 25.9, 24.6, 22.3, 11.6, ppm; ^{11}B NMR (128 MHz, CDCl_3): δ 22.70, 21.34, ppm; HRMS (ESI) calcd for $\text{C}_{27}\text{H}_{40}\text{B}_2\text{N}_3\text{O}_4$ $[\text{M}+\text{H}]^+$ 492.3205, found 492.3214.



diboron-NHC (*N*-heterocyclic carbenes)-adducts B3. give a yellow solid (2.51 g, 50%). ^1H NMR (400 MHz, CDCl_3): δ 8.11 (d, $J = 8.0$ Hz, 1H), 7.56 (t, $J = 8.0$ Hz, 1H), 7.15-7.09 (m, 2H), 6.90 (t, $J = 8.0$ Hz, 1H), 6.79 (t, $J = 8.0$ Hz, 1H), 6.50 (t, $J = 8.0$ Hz, 1H), 6.31 (d, $J = 8.0$ Hz, 1H), 3.30 (t, $J = 8.0$ Hz, 2H), 1.67-1.59 (m, 2H), 1.38-1.34 (m, 2H), 1.22 (d, $J = 4.8$ Hz, 24H), 0.93 (t, $J = 7.6$ Hz, 3H), ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3): δ 159.8, 149.5, 141.8, 139.5, 135.6, 126.1, 117.4, 115.2, 114.5, 110.1, 105.4, 81.7, 47.0, 31.2, 26.1, 25.8, 24.6, 20.6, 14.3, ppm; ^{11}B NMR (128 MHz, CDCl_3): δ 22.77 ppm; HRMS (ESI) calcd for $\text{C}_{28}\text{H}_{42}\text{B}_2\text{N}_3\text{O}_4$ $[\text{M}+\text{H}]^+$ 506.3361, found 506.3360.

5. NMR Spectra for diboron-NHC-adducts B1-B3

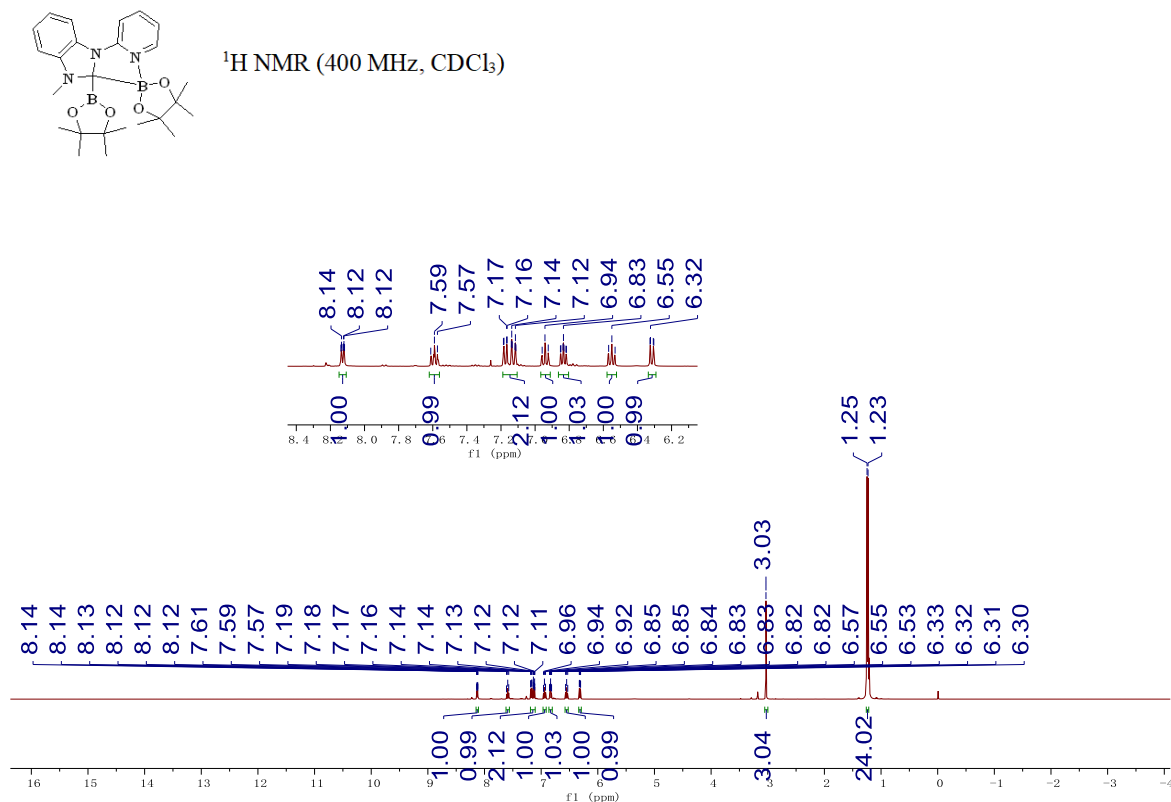


Figure S1. ^1H NMR Spectra of diboron-NHC (*N*-heterocyclic carbenes)-adducts **B1**.

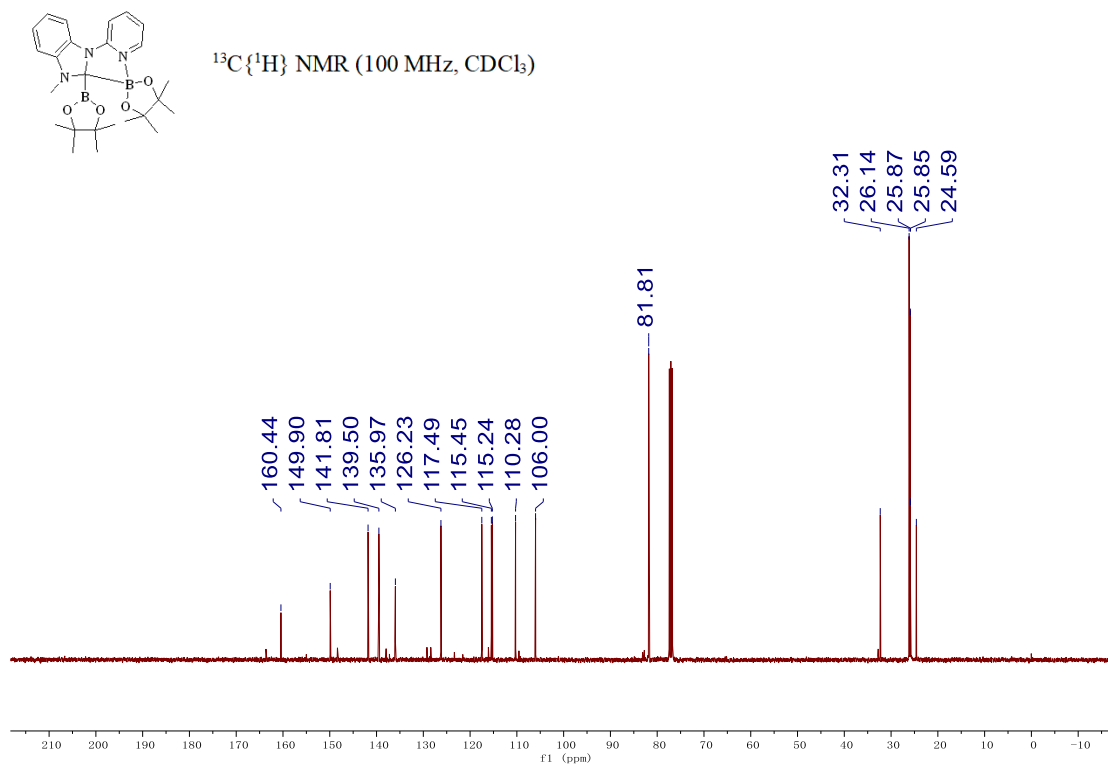


Figure S2. $^{13}\text{C}\{^1\text{H}\}$ NMR Spectra of diboron-NHC (*N*-heterocyclic carbenes)-adducts **B1**.

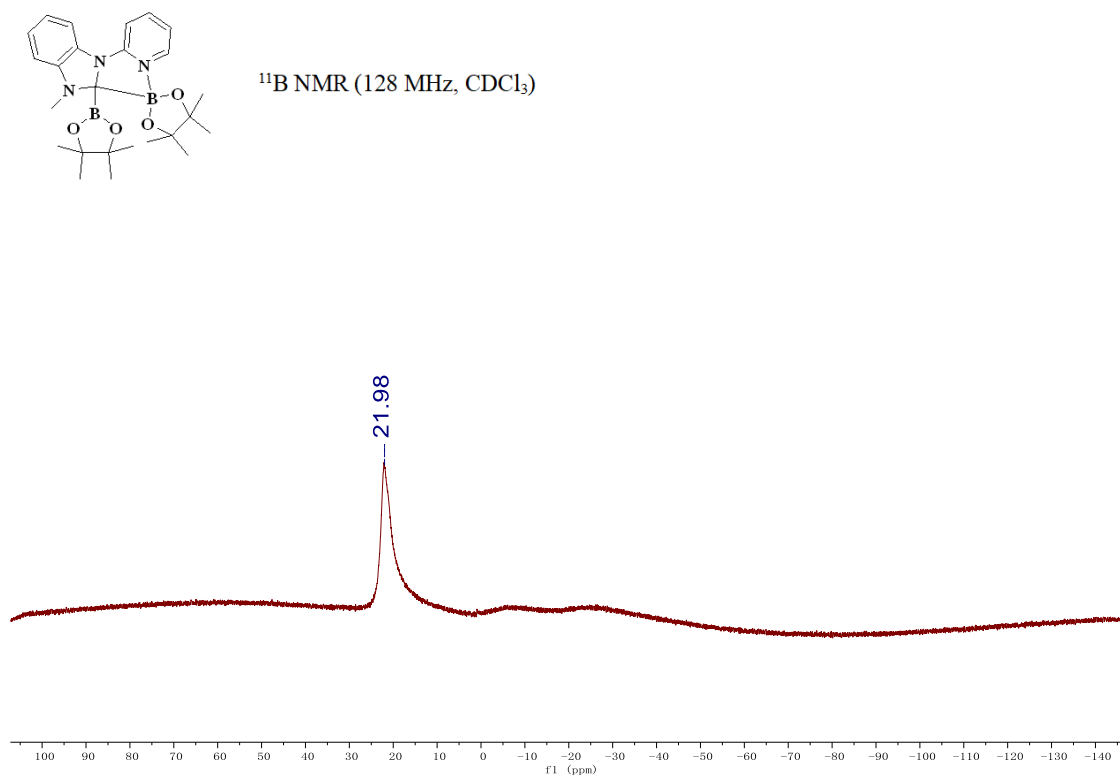


Figure S3. ¹¹B NMR Spectra of diboron-NHC (*N*-heterocyclic carbenes)-adducts **B1**.

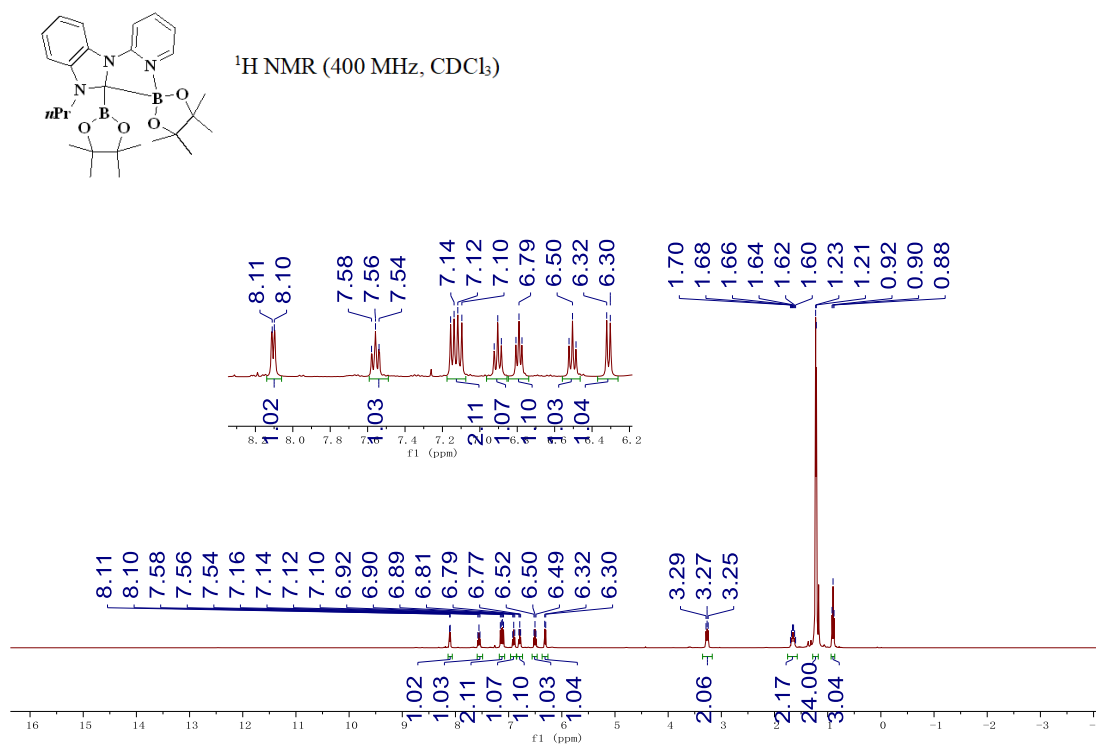


Figure S4. ¹H NMR Spectra of diboron-NHC (*N*-heterocyclic carbenes)-adducts **B2**.

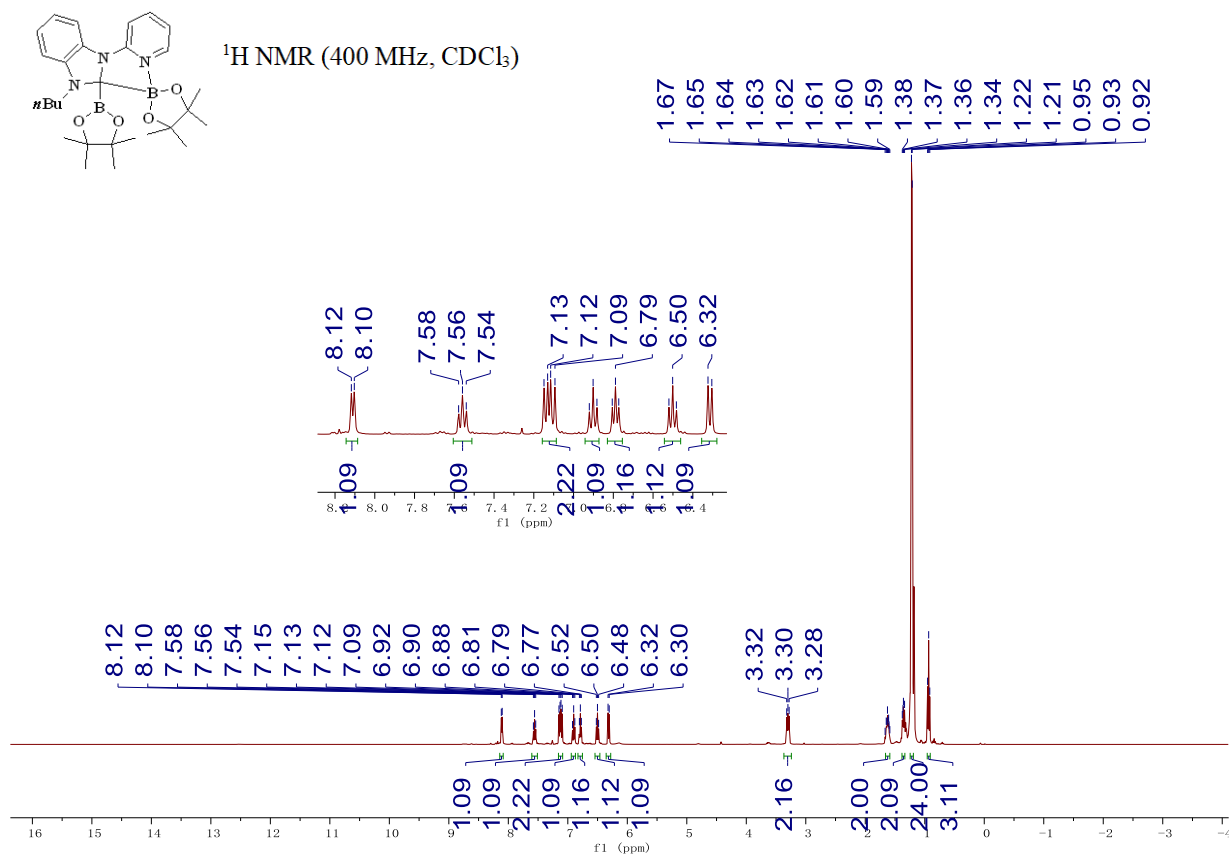


Figure S7. ¹H NMR Spectra of diboron-NHC (*N*-heterocyclic carbenes)-adducts **B3**.

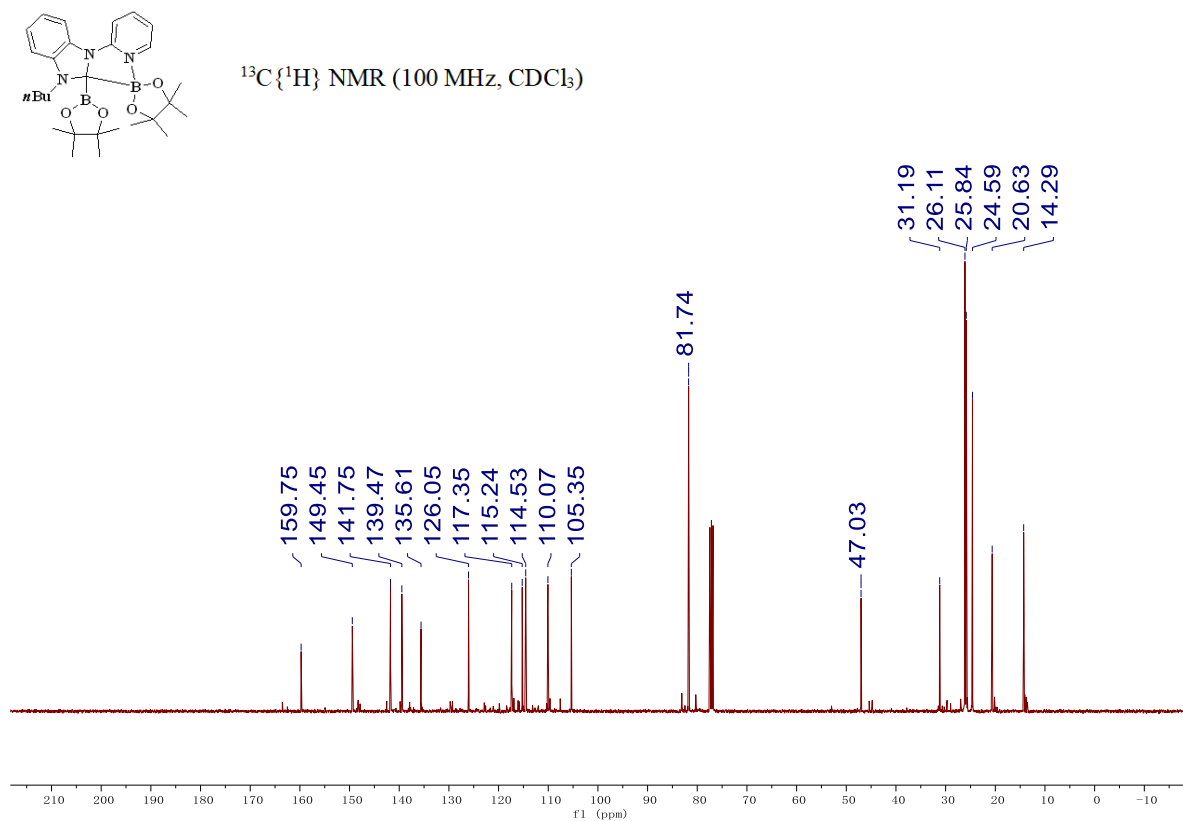
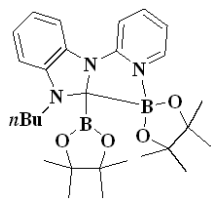


Figure S8. ¹³C{¹H} NMR Spectra of diboron-NHC (*N*-heterocyclic carbenes)-adducts **B3**.



^{11}B NMR (128 MHz, CDCl_3)

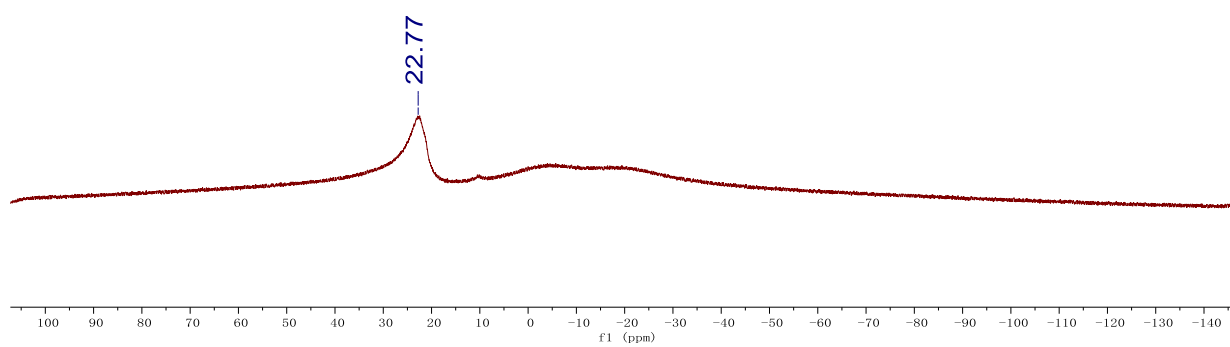
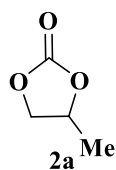
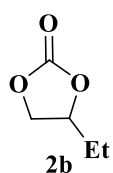


Figure S9. ^{11}B NMR Spectra of diboron-NHC (*N*-heterocyclic carbenes)-adducts **B3**.

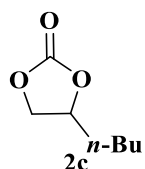
6. Characterization Data for the Cyclic Carbonate



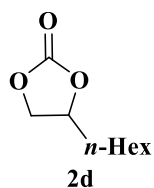
4-methyl-1,3-dioxolan-2-one (2a).² Purification by flash chromatography (petroleum ether: EtOAc = 2:1) give a colorless oil (1.1257 g, 97%). ¹H NMR (400 MHz, CDCl₃): δ 4.82-4.77 (m, 1H), 4.49 (t, *J* = 8.0 Hz, 1H), 3.95 (dd, *J* = 8.0 Hz, *J* = 4.0 Hz, 1H), 1.41-1.39 (m, 3H) ppm. ¹³C{¹H} NMR (100 MHz, CDCl₃): δ 155.0 (C=O), 73.6 (CH), 70.6 (CH₂), 19.1 (CH₃), ppm.



4-ethyl-1,3-dioxolan-2-one (2b).³ Purification by flash chromatography (petroleum ether: EtOAc = 2:1) give a yellow oil (1.1489 g, 99%). ¹H NMR (400 MHz, CDCl₃): δ 4.63-4.58 (m, 1H), 4.47 (t, *J* = 8.0 Hz, 1H), 4.02 (dd, *J* = 8.0 Hz, *J* = 8.0 Hz, 1H), 1.77-1.64 (m, 2H), 0.94 (t, *J* = 7.6 Hz, 3H) ppm. ¹³C{¹H} NMR (100 MHz, CDCl₃): δ 155.2 (C=O), 78.1 (CH), 69.0 (CH₂), 26.8 (CH₂), 8.3 (CH₃), ppm.

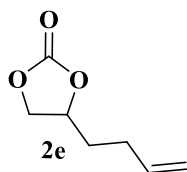


4-butyl-1,3-dioxolan-2-one (2c).³ Purification by flash chromatography (petroleum ether: EtOAc = 2:1) give a colorless oil (1.0909 g, 94%). ¹H NMR (400 MHz, CDCl₃): δ 4.72-4.65 (m, 1H), 4.51 (t, *J* = 8.0 Hz, 1H), 4.05 (t, *J* = 8.0 Hz, 1H), 1.80-1.64 (m, 2H), 1.46-1.31 (m, 4H), 0.90 (t, *J* = 8.0 Hz, 3H). ¹³C{¹H} NMR (100 MHz, CDCl₃): δ 155.2 (C=O), 77.1 (CH), 69.4 (CH₂), 33.6 (CH₂), 26.4 (CH₂), 22.3 (CH₂), 13.8 (CH₃), ppm.

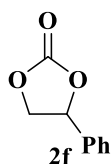


4-hexyl-1,3-dioxolan-2-one (2d).⁴ Purification by flash chromatography (petroleum ether: EtOAc = 5:1): have a colorless oil (1.0793 g, 93%). ¹H NMR (400 MHz, CDCl₃):

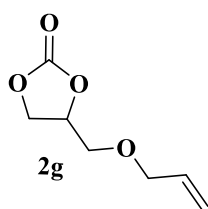
δ 4.70-4.63 (m, 1H), 4.49 (t, J = 8.0 Hz, 1H), 4.04-4.01 (m, 1H), 1.80-1.71 (m, 1H), 1.67-1.61 (m, 1H), 1.43-1.25 (m, 8H), 0.86-0.83 (m, 3H). ppm. $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3): δ 155.2 (C=O), 77.2 (CH), 69.5 (CH_2), 33.8 (CH_2), 31.5 (CH_2), 28.8 (CH_2), 24.3 (CH_2), 22.4 (CH_2), 14.0 (CH_3), ppm.



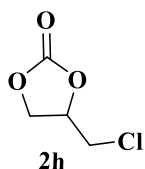
4-(but-3-en-1-yl)-1,3-dioxolan-2-one (2e).⁵ Purification by flash chromatography (petroleum ether: EtOAc = 2:1): gave a colorless oil (1.1257 g, 97%). ^1H NMR (400 MHz, CDCl_3): δ 5.80-5.70 (m, 1H), 5.07-4.99 (m, 2H), 4.73-4.66 (m, 1H), 4.50 (t, J = 8.0 Hz, 1H), 4.05 (t, J = 8.0 Hz, 1H), 2.26-2.09 (m, 2H), 1.93-1.84 (m, 1H), 1.79-1.70 (m, 1H) ppm. $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3): δ 155.0 (C=O), 136.2 (CH), 116.3 (CH_2), 76.4 (CH), 69.4 (CH_2), 33.0 (CH_2), 28.6 (CH_2), ppm.



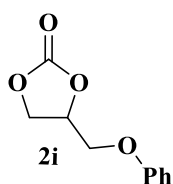
4-phenyl-1,3-dioxolan-2-one (2f).² Purification by flash chromatography (petroleum ether: EtOAc = 2:1): gave a white solid (1.1025 g, 95%), melting point (MP) = 50.0-51.4 °C. ^1H NMR (400 MHz, CDCl_3): δ 7.41-7.39 (m, 3H), 7.34-7.33 (m, 2H), 5.65 (t, J = 8.0 Hz, 1H), 4.77 (t, J = 8.0 Hz, 1H), 4.29 (t, J = 8.0 Hz, 1H). ppm. $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3): δ 154.9 (C=O), 135.8 (C), 129.5 (CH), 129.0 (2 \times CH), 125.9 (2 \times CH), 77.9 (CH), 71.0 (CH_2), ppm.



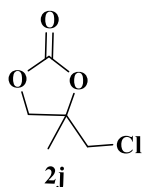
4-((allyloxy)methyl)-1,3-dioxolan-2-one (2g).⁵ Purification by flash chromatography (petroleum ether: EtOAc = 2:1): gave a colorless oil (1.0909 g, 94%). ^1H NMR (400 MHz, CDCl_3): δ 5.84-5.74 (m, 1H), 5.23-5.11 (m, 2H), 4.80-4.75 (m, 1H), 4.44 (t, J = 8.0 Hz, 1H), 4.32-4.28 (m, 1H), 3.98-3.96 (m, 2H), 3.63 (dd, J = 12.0 Hz, J = 4.0 Hz, 1H), 3.55-3.51 (m, 1H) ppm. $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3): δ 155.0 (C=O), 133.7 (CH), 117.5 (CH_2), 75.2 (CH), 72.3 (CH_2), 68.8 (CH_2), 66.1 (CH_2), ppm.



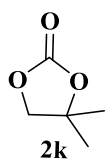
4-(chloromethyl)-1,3-dioxolan-2-one (2h).³ Purification by flash chromatography (petroleum ether: EtOAc = 2:1): gave a yellow oil (1.0445 g, 90%). ¹H NMR (400 MHz, CDCl₃): δ 5.00-4.95 (m, 1H), 4.54 (t, J = 8.8 Hz, 1H), 4.34 (dd, J = 8.0 Hz, J = 4.0 Hz, 1H), 3.79 (dd, J = 12.0 Hz, J = 4.0 Hz, 1H), 3.68 (dd, J = 12.0 Hz, J = 4.0 Hz, 1H). ppm. ¹³C{¹H} NMR (100 MHz, CDCl₃): δ 154.5 (C=O), 74.5 (CH), 66.9 (CH₂), 44.1 (CH₂), ppm.



4-(phenoxymethyl)-1,3-dioxolan-2-one (2i).³ Purification by flash chromatography (petroleum ether : EtOAc = 2:1): gave a white solid (1.0561 g, 91%), MP = 100.9-101.2 °C. ¹H NMR (400 MHz, CDCl₃): δ 7.33-7.29 (m, 2H), 7.04-6.99 (m, 1H), 6.92-6.90 (m, 2H), 5.05-4.99 (m, 1H), 4.60 (t, J = 8.0 Hz, 1H), 4.52 (dd, J = 8.0 Hz, J = 8.0 Hz, 1H), 4.23 (dd, J = 12.0 Hz, J = 4.0 Hz, 1H), 4.13 (dd, J = 10.8 Hz, J = 3.6 Hz, 1H). ppm. ¹³C{¹H} NMR (100 MHz, CDCl₃): δ 157.9 (C=O), 154.8 (C), 129.8 (2×CH), 122.1 (CH), 114.7 (2×CH), 74.3 (CH), 67.0 (CH₂), 66.3 (CH₂), ppm.

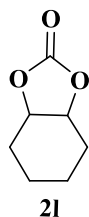


4-(chloromethyl)-4-methyl-1,3-dioxolan-2-one (2j).⁶ Purification by flash chromatography (petroleum ether : EtOAc = 5:1): gave a colorless oil (0.9052 g, 78%). ¹H NMR (400 MHz, CDCl₃): δ 4.46-4.43 (m, 1H), 4.13 (d, J = 8.8 Hz, 1H), 3.69 (d, J = 12.0 Hz, 1H), 3.58 (d, J = 12.0 Hz, 1H), 1.56 (s, 3H), ppm. ¹³C{¹H} NMR (100 MHz, CDCl₃): δ 153.9, 81.8, 71.9, 48.5, 22.9, ppm.

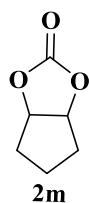


4,4-dimethyl-1,3-dioxolan-2-one (2k).⁷ Purification by flash chromatography

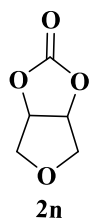
(petroleum ether: EtOAc = 2:1) gave a colorless oil (0.8704 g, 75%). ^1H NMR (400 MHz, CDCl_3): δ 4.12 (s, 2H), 1.49 (s, 6H) ppm. $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3): δ 154.7 (C=O), 81.8 (C), 75.4 (CH_2), 26.0 ($2\times\text{CH}_3$), ppm.



hexahydrobenzo [d] [1, 3] dioxol-2-one (2l).² Purification by flash chromatography (petroleum ether: EtOAc = 2:1): Colorless oil (0.8704 g, 75%). ^1H NMR (400 MHz, CDCl_3): δ 4.68-4.63 (m, 2H), 1.87-1.82 (m, 4H), 1.59-1.54 (m, 2H), 1.42-1.37 (m, 2H) ppm. $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3): δ 155.4 (C=O), 75.8 ($2\times\text{CH}$), 26.7 ($2\times\text{CH}_2$), 19.1 ($2\times\text{CH}_2$), ppm.



tetrahydro-4H-cyclopenta[d] [1, 3] dioxol-2-one (2m).⁷ Purification by flash chromatography (petroleum ether: EtOAc = 2:1) gave a white solid (0.8588 g, 74%). ^1H NMR (400 MHz, CDCl_3): δ 5.05-5.04 (m, 2H), 2.05-2.01 (m, 2H), 1.75-1.56 (m, 4H), ppm. $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3): δ 155.4 (C=O), 81.8 ($2\times\text{CH}$), 32.9 ($2\times\text{CH}_2$), 21.4 (CH_2), ppm.



tetrahydrofuro[3,4-d] [1, 3] dioxol-2-one (2n).⁷ Purification by flash chromatography (petroleum ether: EtOAc = 2:1) gave a white solid (0.9052 g, 78%). ^1H NMR (400 MHz, CDCl_3): δ 5.19-5.15 (m, 2H), 4.16-4.13 (m, 2H), 3.52-3.49 (m, 2H) ppm. $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3): δ 154.6 (C=O), 80.2 ($2\times\text{CH}$), 73.0 ($2\times\text{CH}_2$), ppm.

7. NMR Spectra for the Cyclic Carbonates

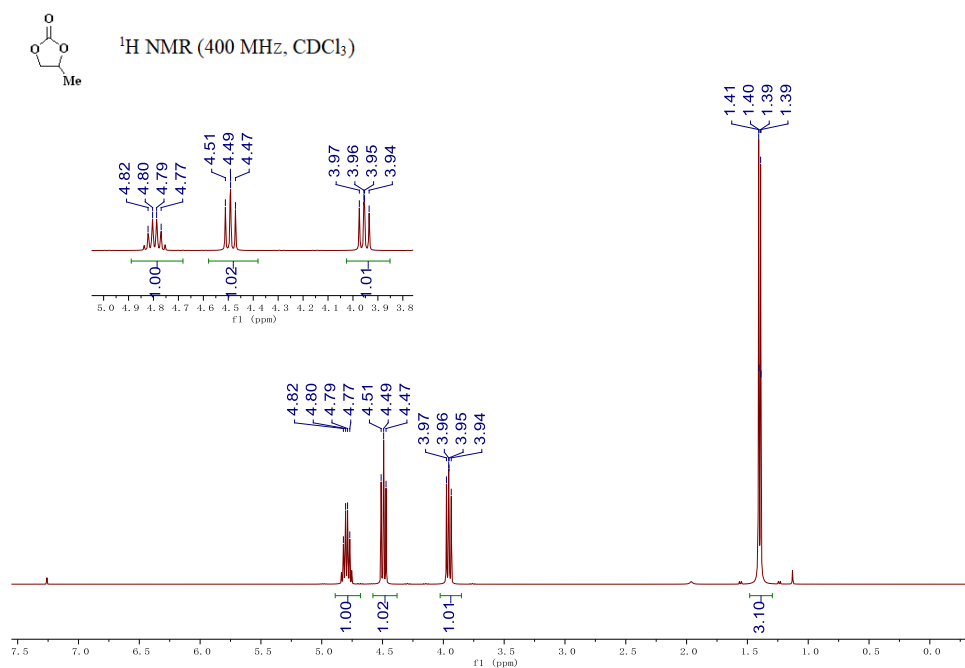


Figure S10. ^1H NMR Spectra of 4-Methyl-1,3-dioxolan-2-one (2a).

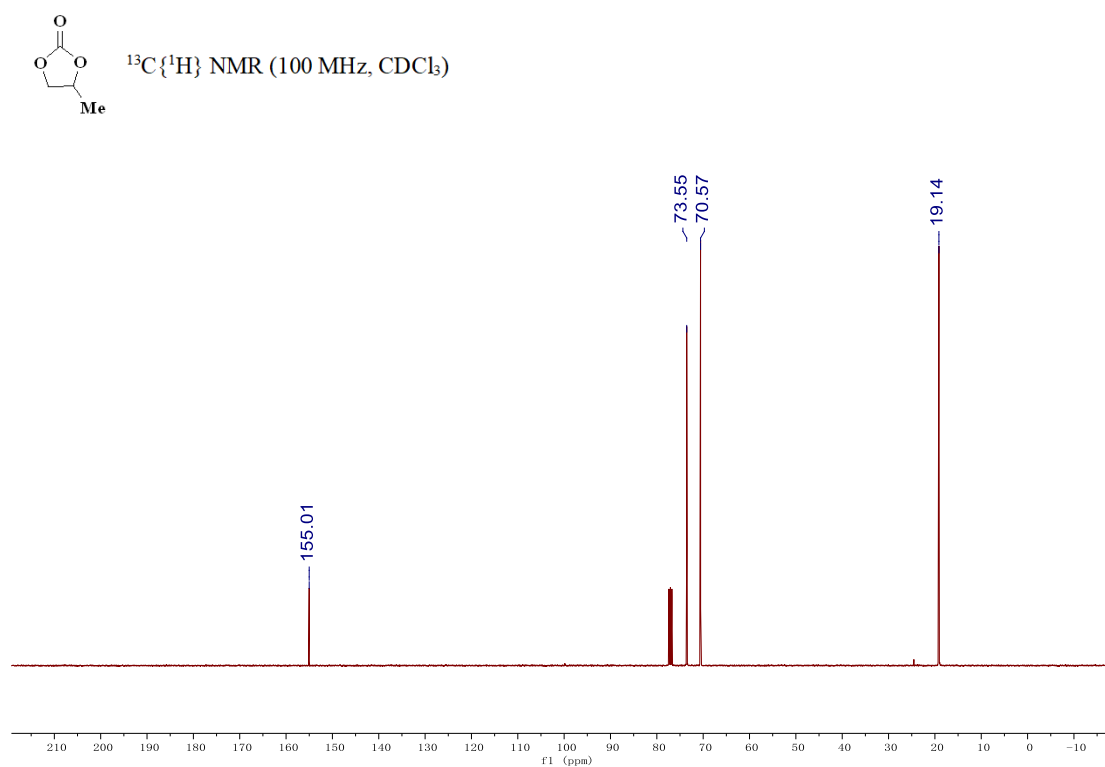


Figure S11. $^{13}\text{C}\{^1\text{H}\}$ NMR Spectra of 4-Methyl-1,3-dioxolan-2-one (2a).

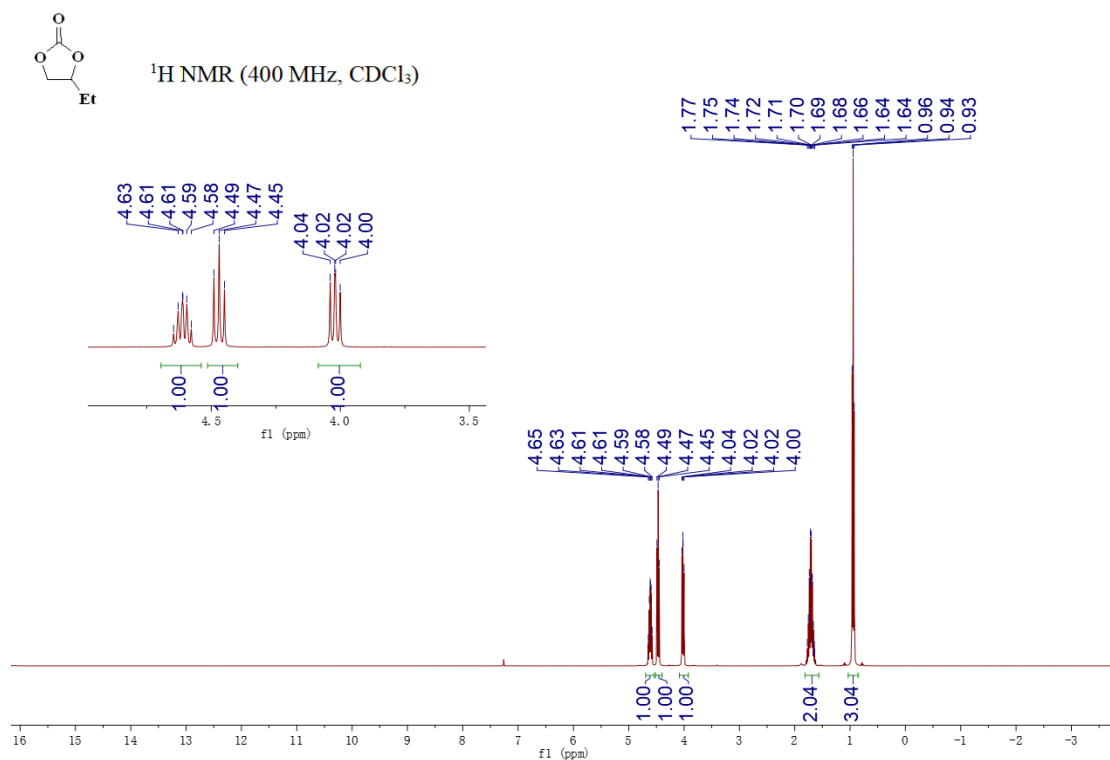


Figure S12. ¹H NMR Spectra of 4-Ethyl-1,3-dioxolan-2-one (2b).

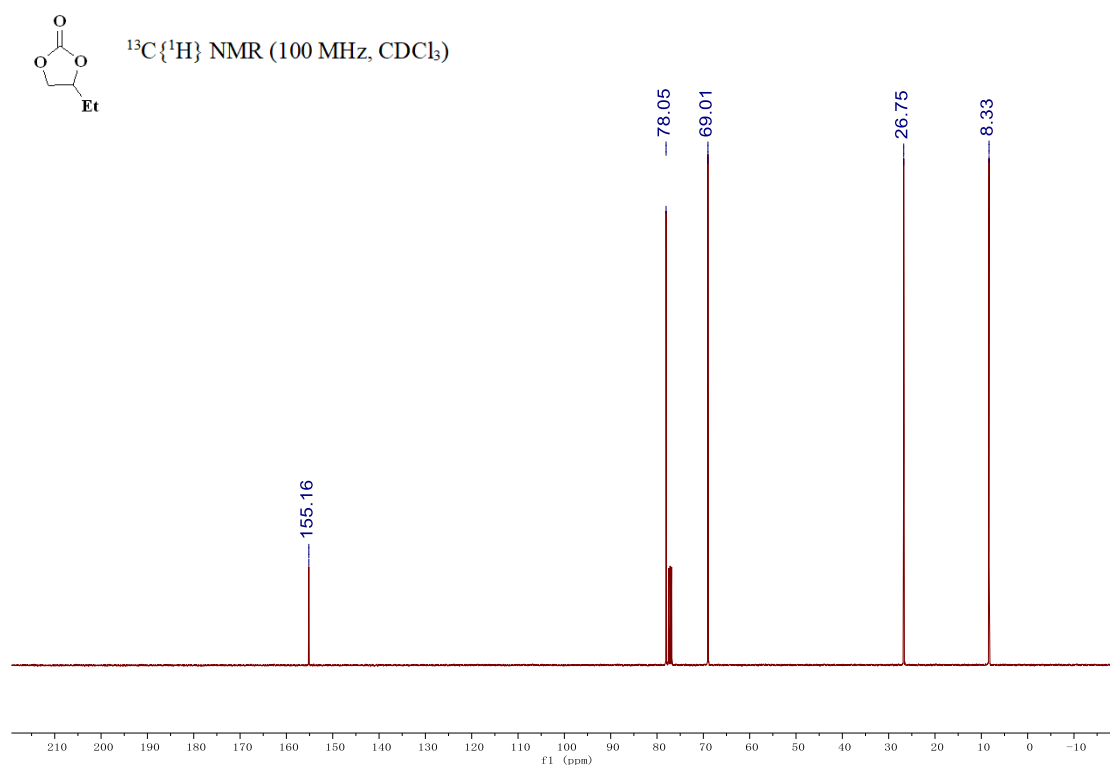


Figure S13. ¹³C{¹H} NMR Spectra of 4-Ethyl-1,3-dioxolan-2-one (2b).

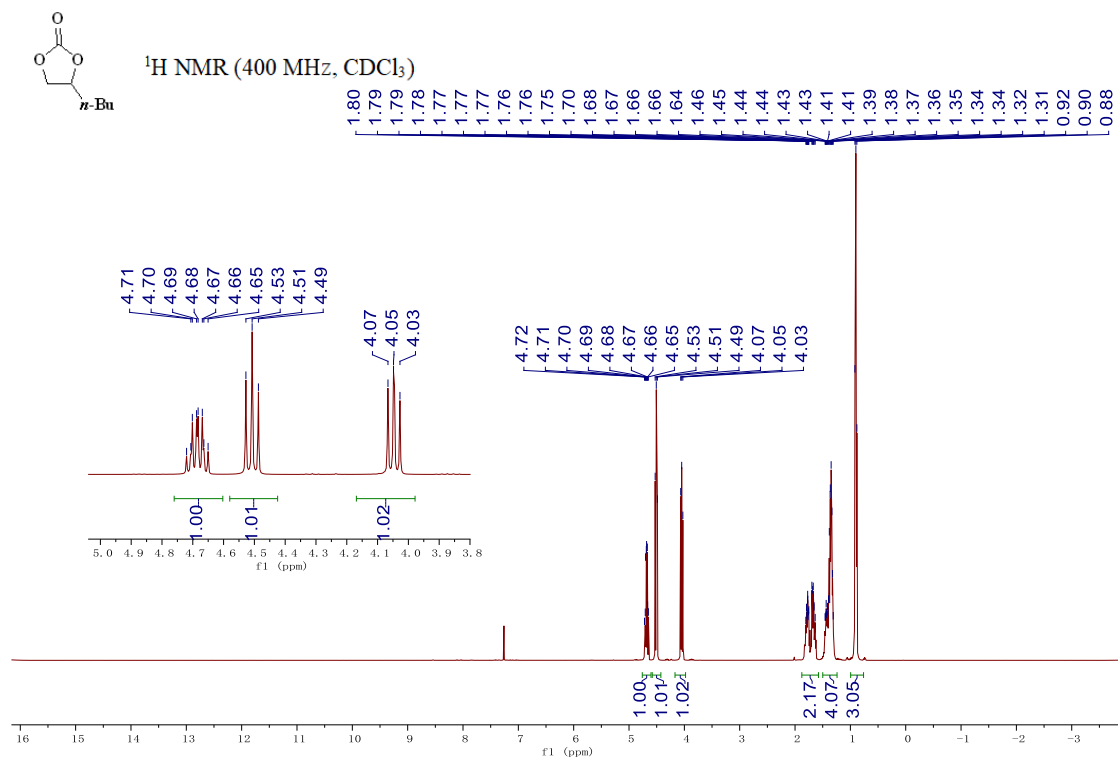


Figure S14. ¹H NMR Spectra of 4-Butyl-1,3-dioxolan-2-one (2c).

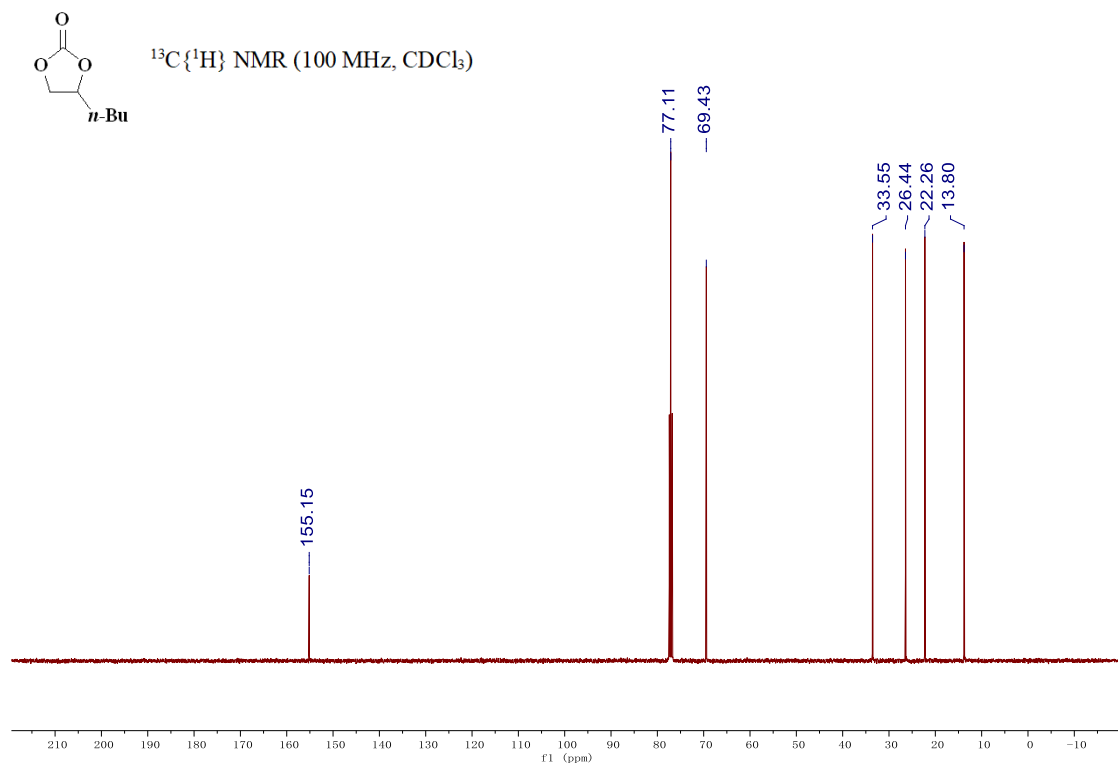


Figure S15. ¹³C{¹H} NMR Spectra of 4-Butyl-1,3-dioxolan-2-one (2c).

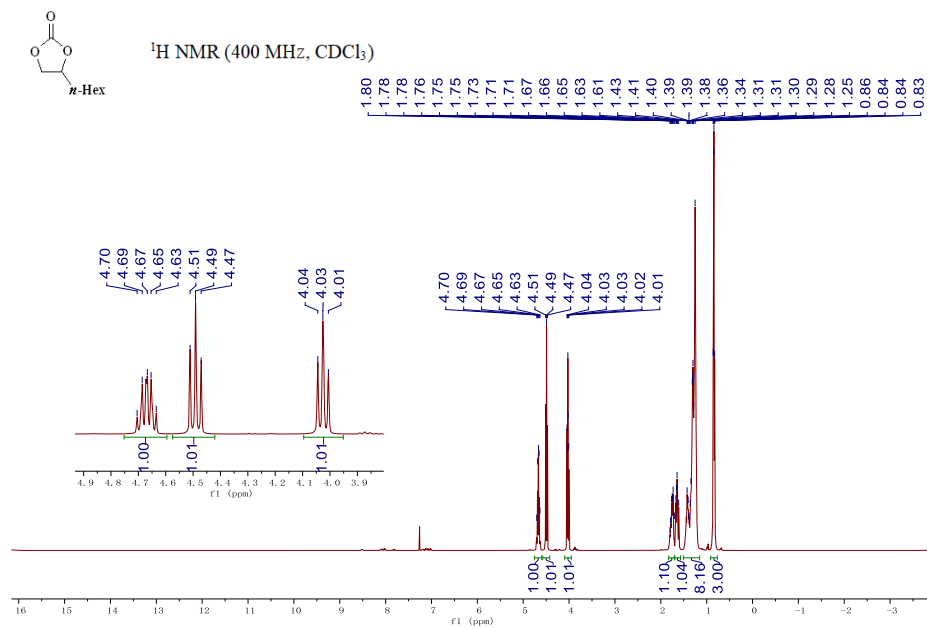


Figure S16. ^1H NMR Spectra of 4-Hexyl-1,3-dioxolan-2-one (2d).

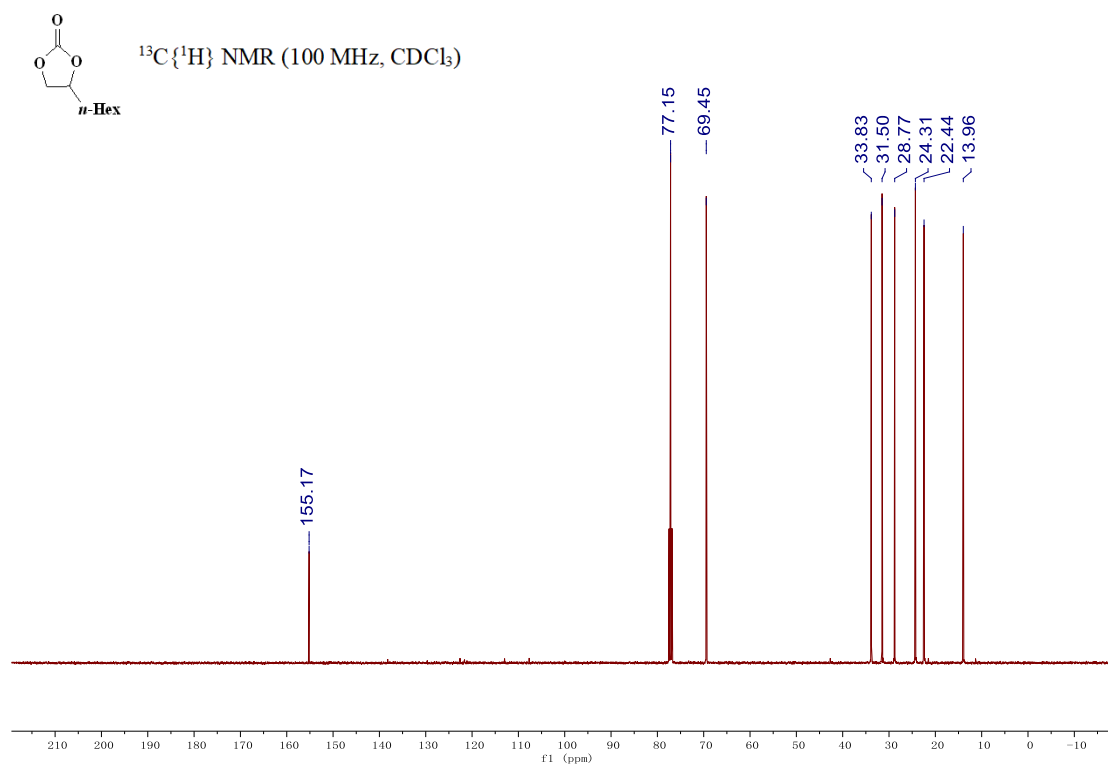


Figure S17. $^{13}\text{C}\{^1\text{H}\}$ NMR Spectra of 4-Hexyl-1,3-dioxolan-2-one (2d).

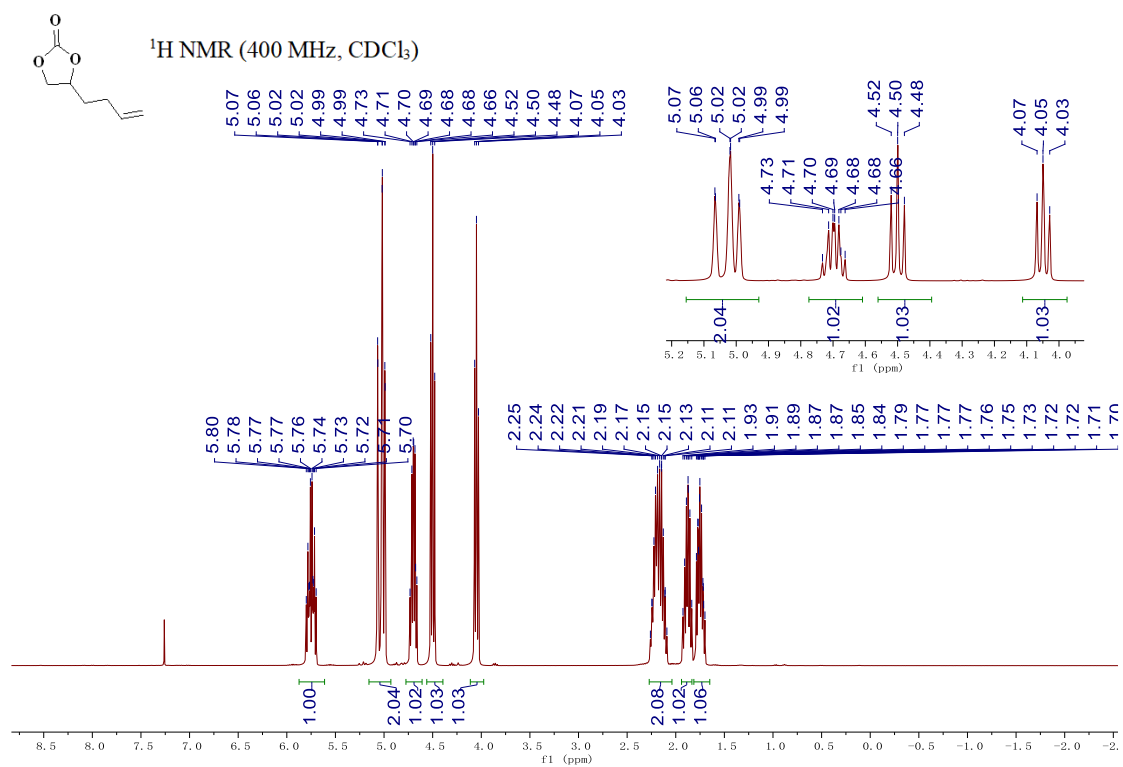


Figure S18. $^1\text{H NMR}$ Spectra of 4-(But-3-en-1-yl)-1,3-dioxolan-2-one (2e).

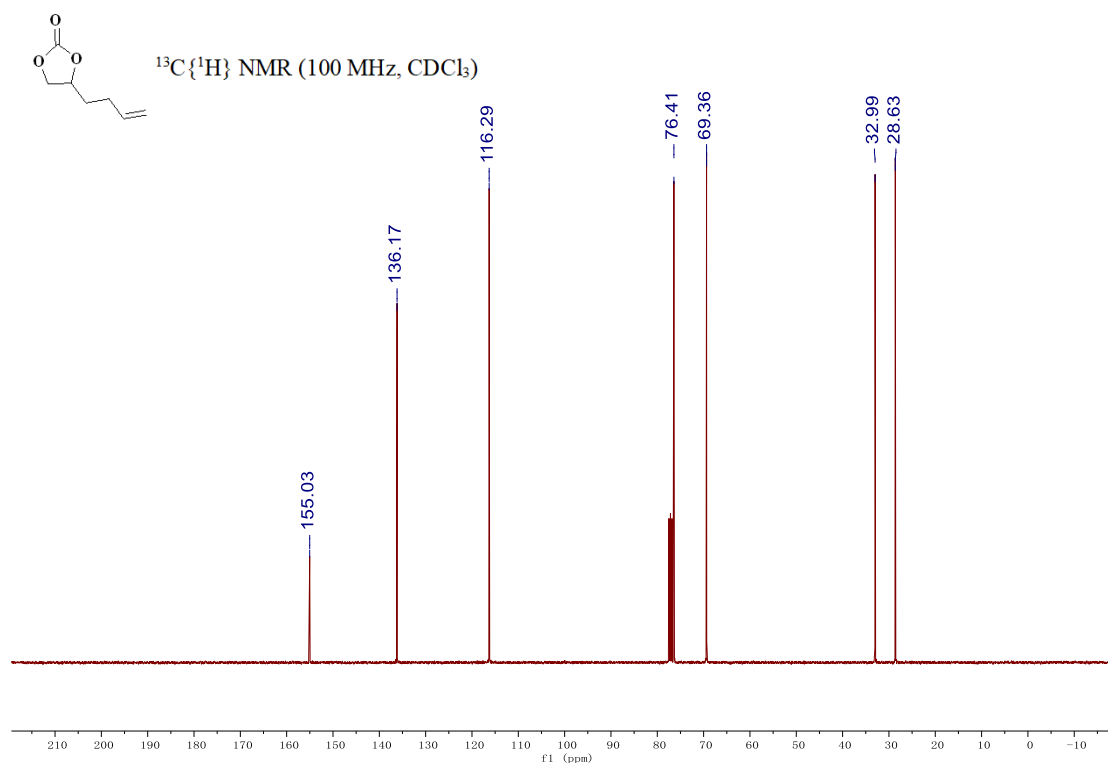


Figure S19. $^{13}\text{C}\{^1\text{H}\}$ NMR Spectra of 4-(But-3-en-1-yl)-1,3-dioxolan-2-one (2e).

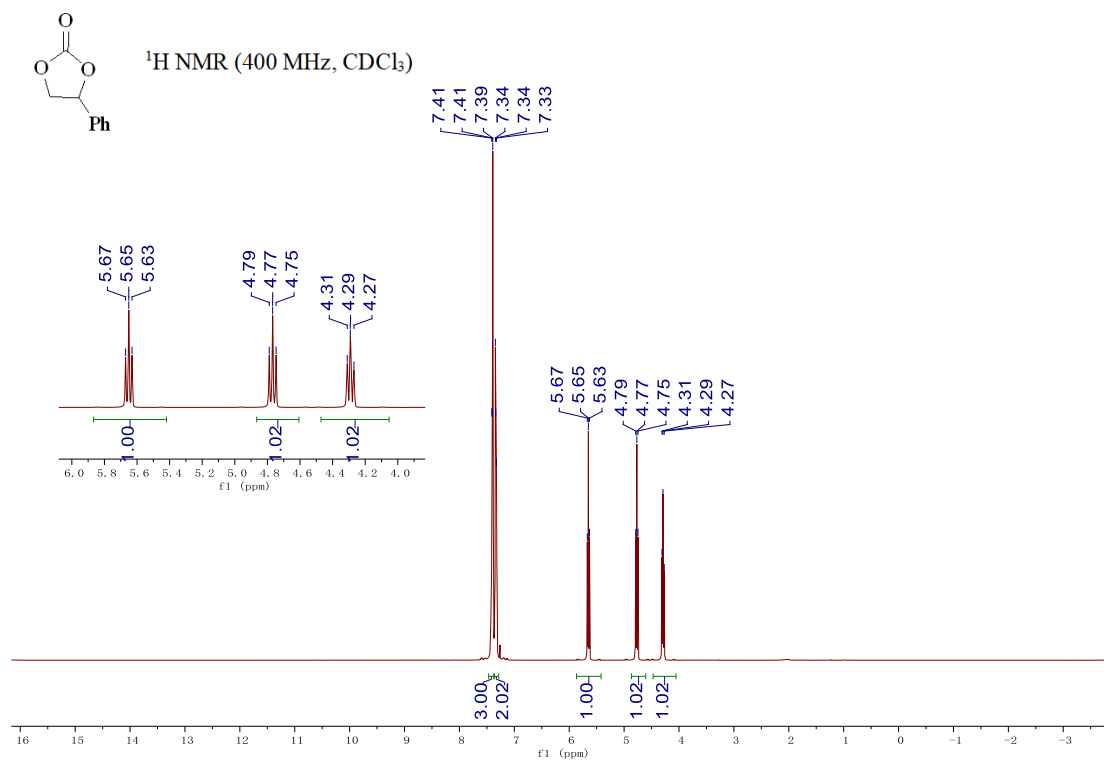


Figure S20. ^1H NMR Spectra of 4-Phenyl-1,3-dioxolan-2-one (2f).

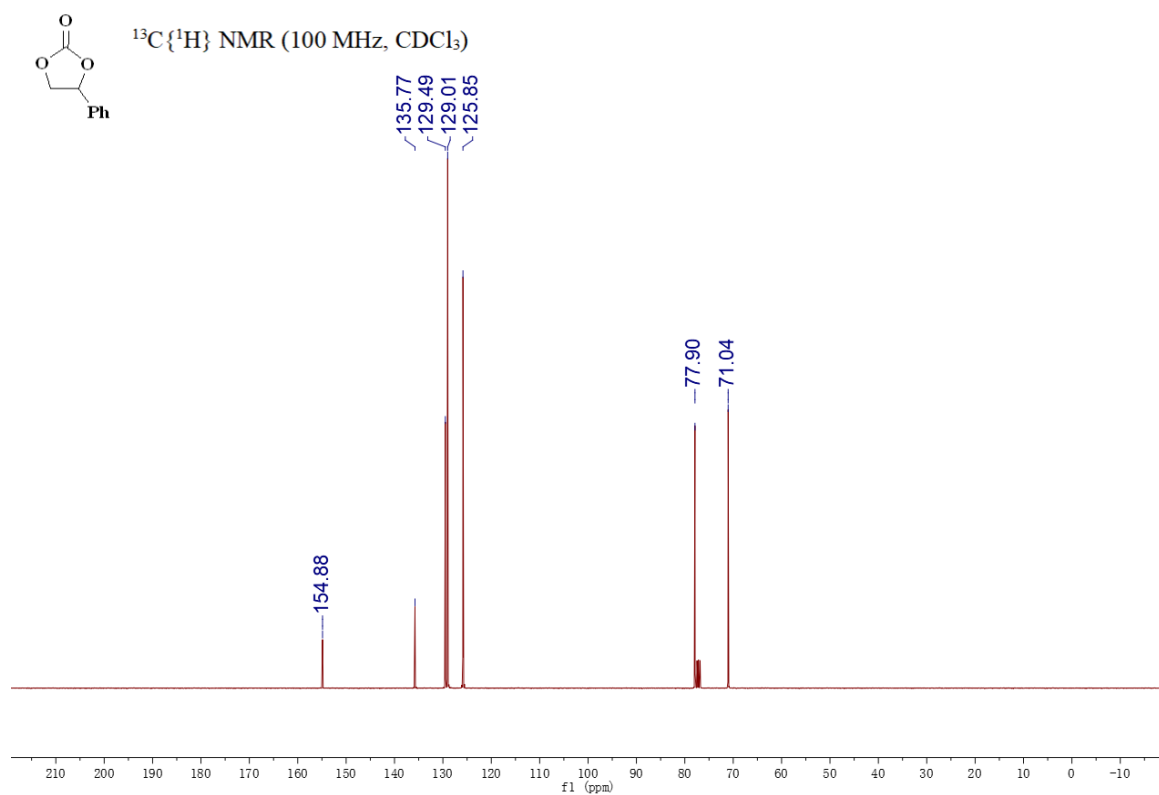


Figure S21. $^{13}\text{C}\{^1\text{H}\}$ NMR Spectra of 4-Phenyl-1,3-dioxolan-2-one (2f).

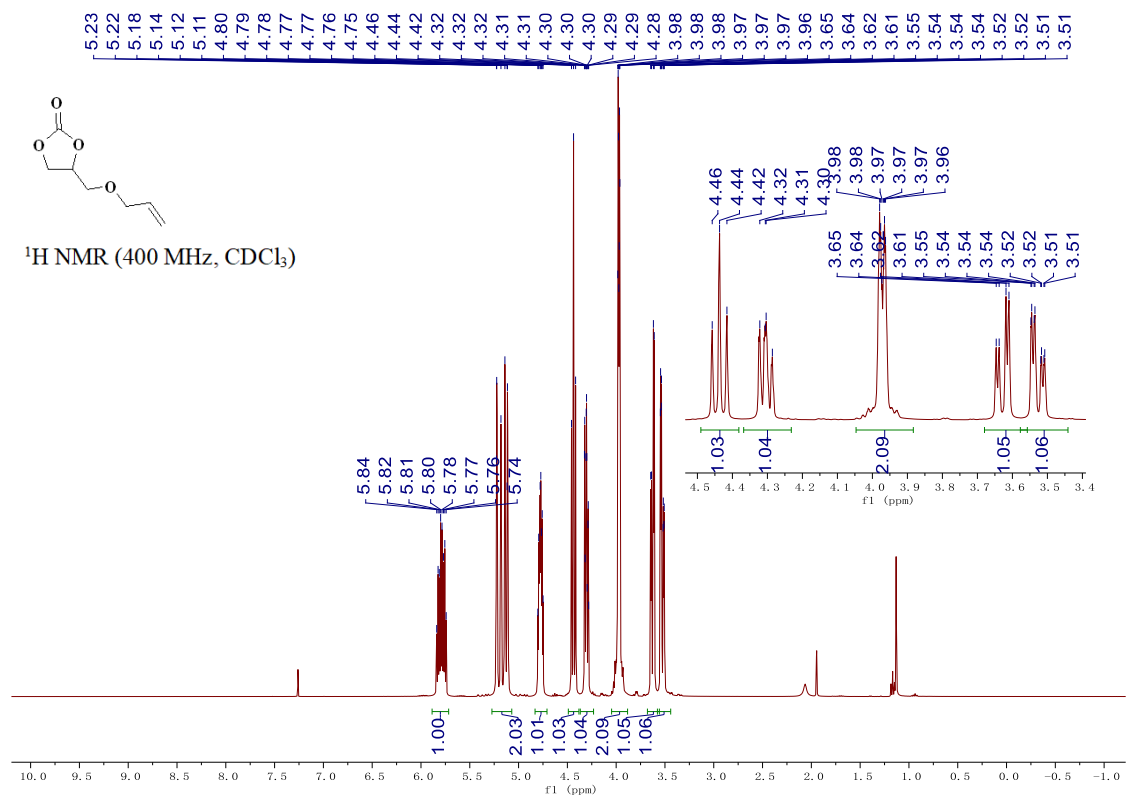


Figure S22. ¹H NMR Spectra of 4-((Allyloxy)methyl)-1,3-dioxolan-2-one (2g).

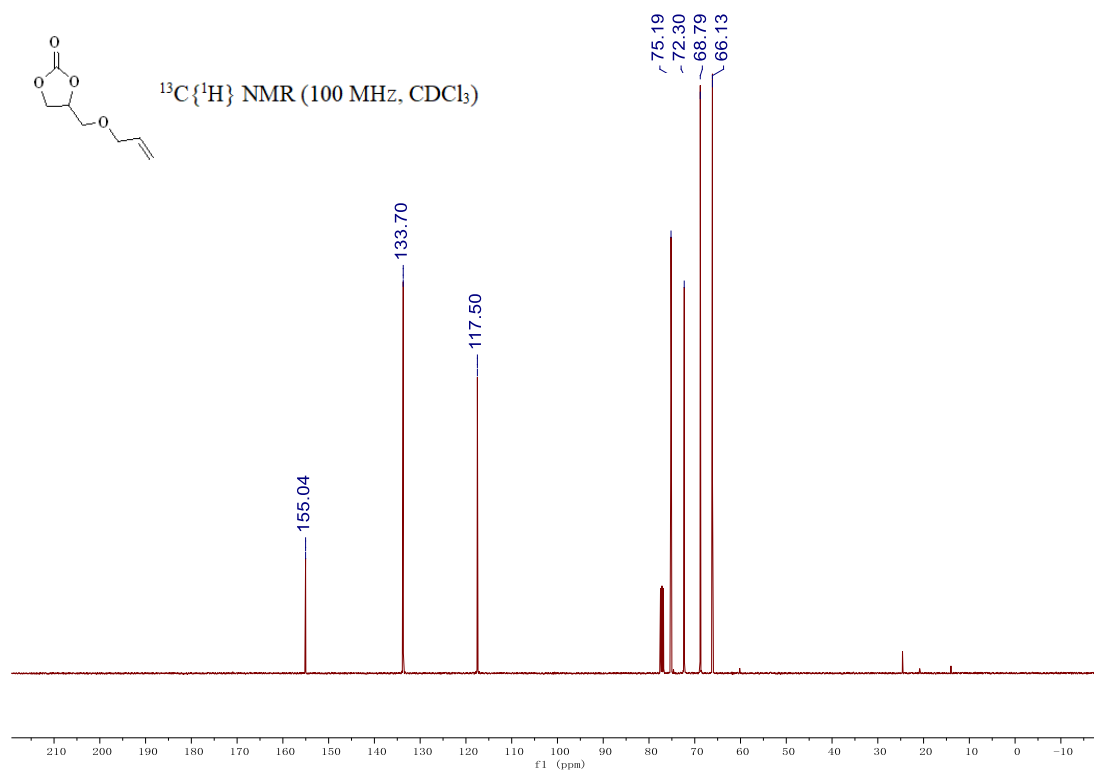


Figure S23. ¹³C{¹H} NMR Spectra of 4-((Allyloxy)methyl)-1,3-dioxolan-2-one (2g).

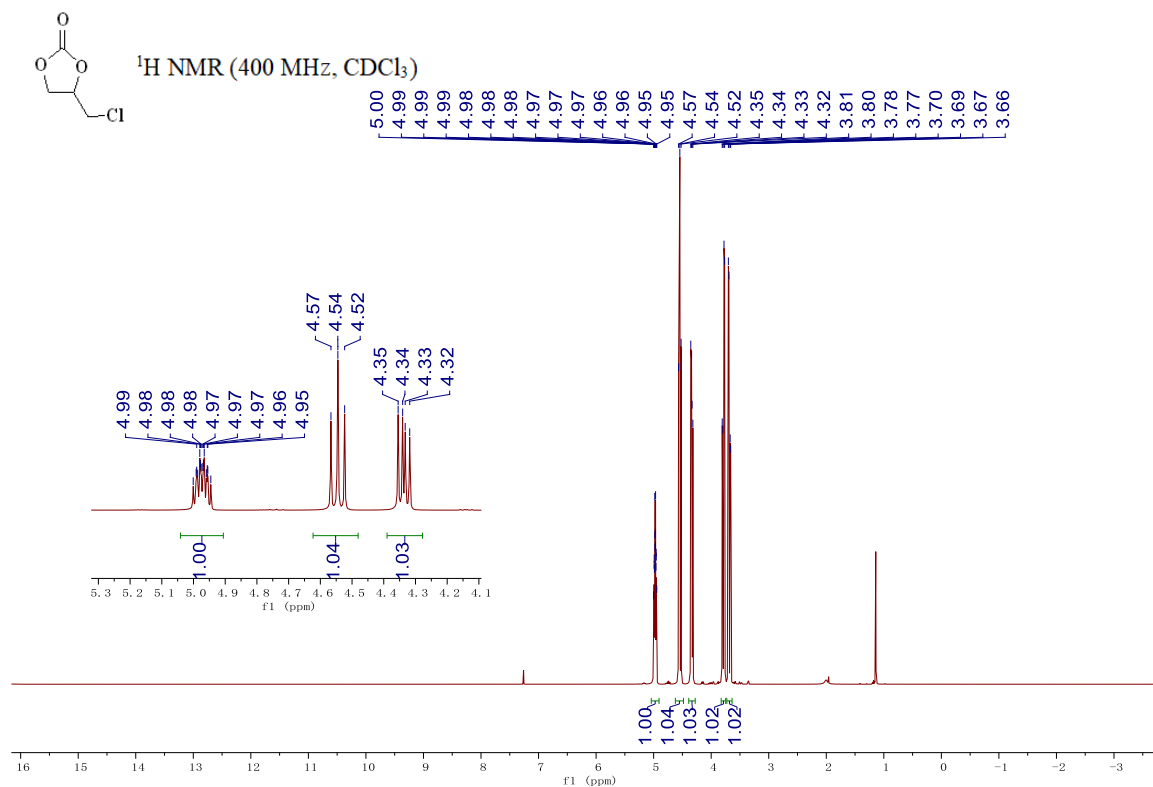


Figure S24. ¹H NMR Spectra of 4-(Chloromethyl)-1,3-dioxolan-2-one (2h).

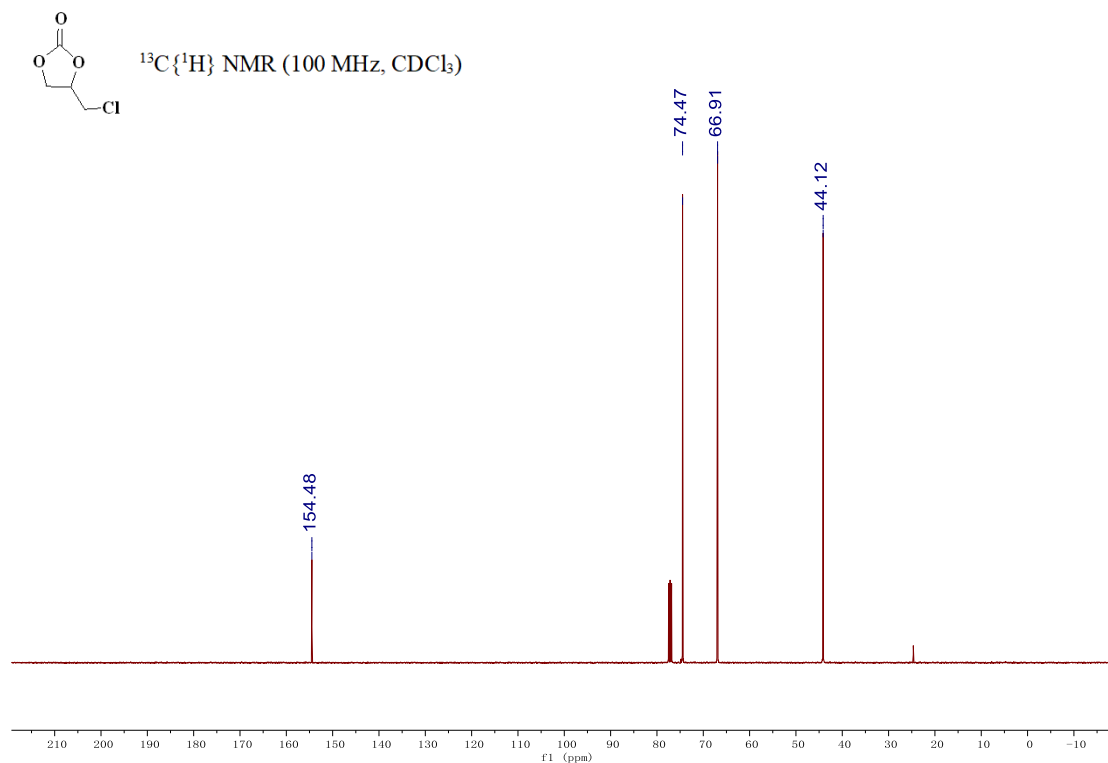


Figure S25. ¹³C{¹H} NMR Spectra of 4-(Chloromethyl)-1,3-dioxolan-2-one (2h).

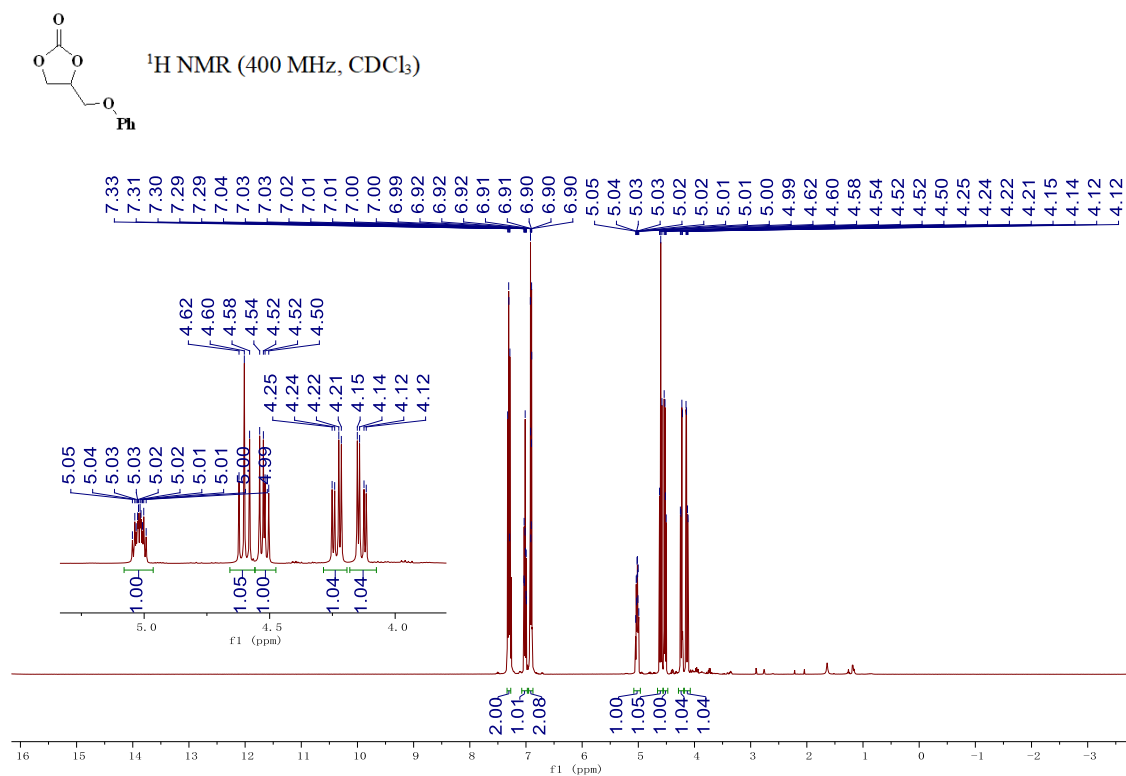


Figure S26. ¹H NMR Spectra of 4-(Phenoxymethyl)-1,3-dioxolan-2-one (2i).

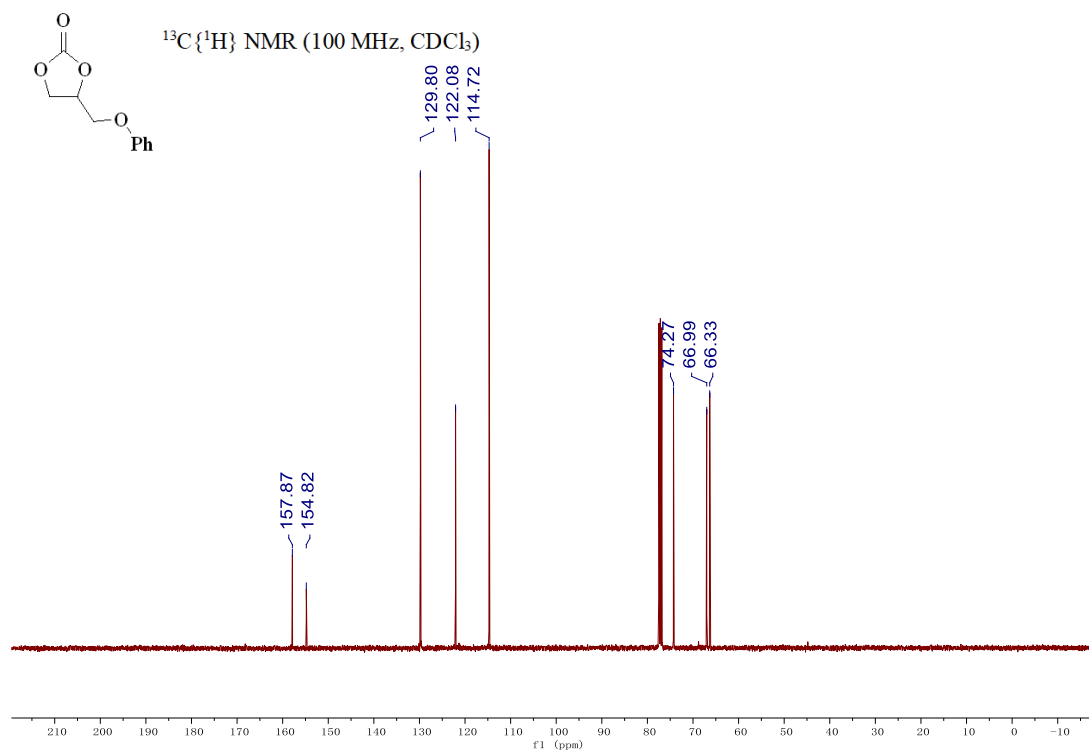


Figure S27. ¹³C{¹H} NMR Spectra of 4-(Phenoxymethyl)-1,3-dioxolan-2-one (2i).

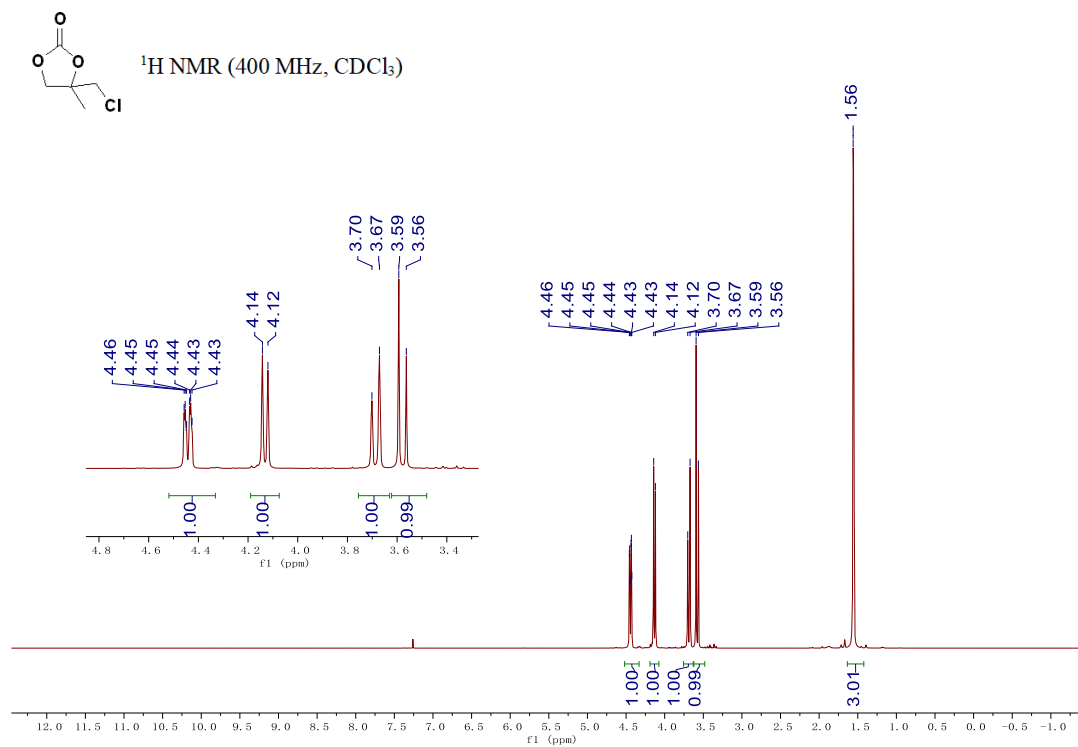


Figure S28. ¹H NMR Spectra of 4-(chloromethyl)-4-methyl-1,3-dioxolan-2-one (2j).

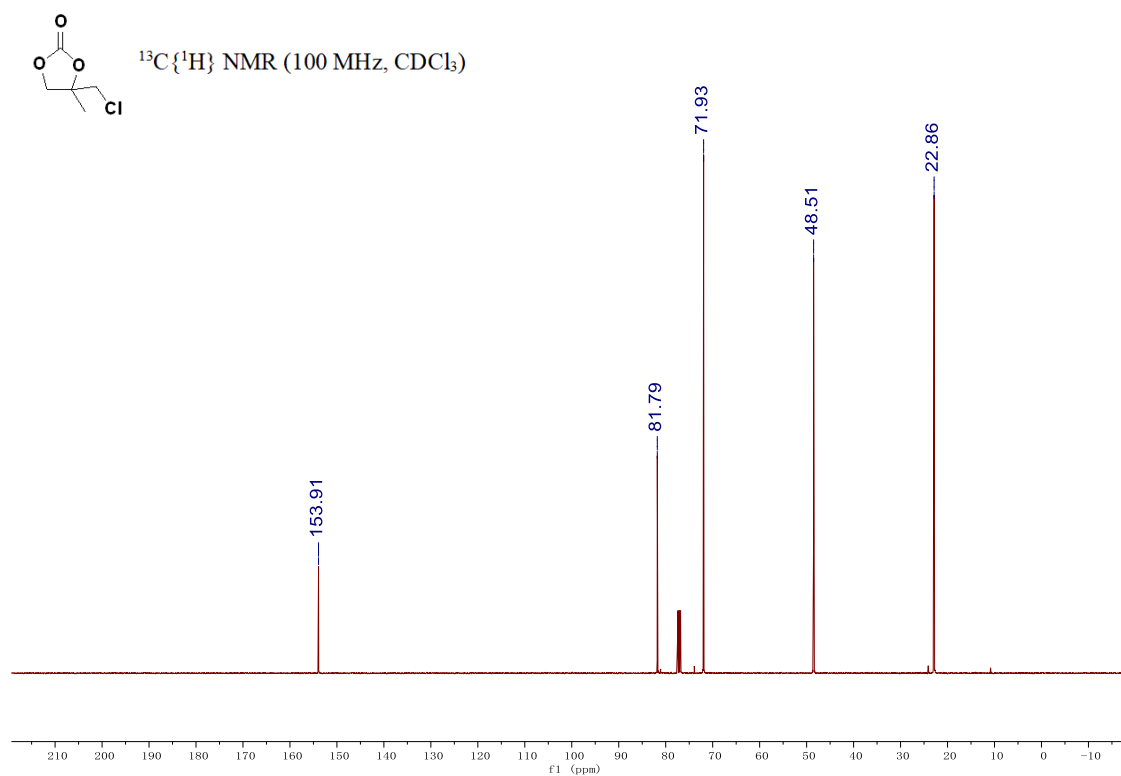


Figure S29. ¹³C{¹H} NMR Spectra of 4-(chloromethyl)-4-methyl-1,3-dioxolan-2-one (2j).

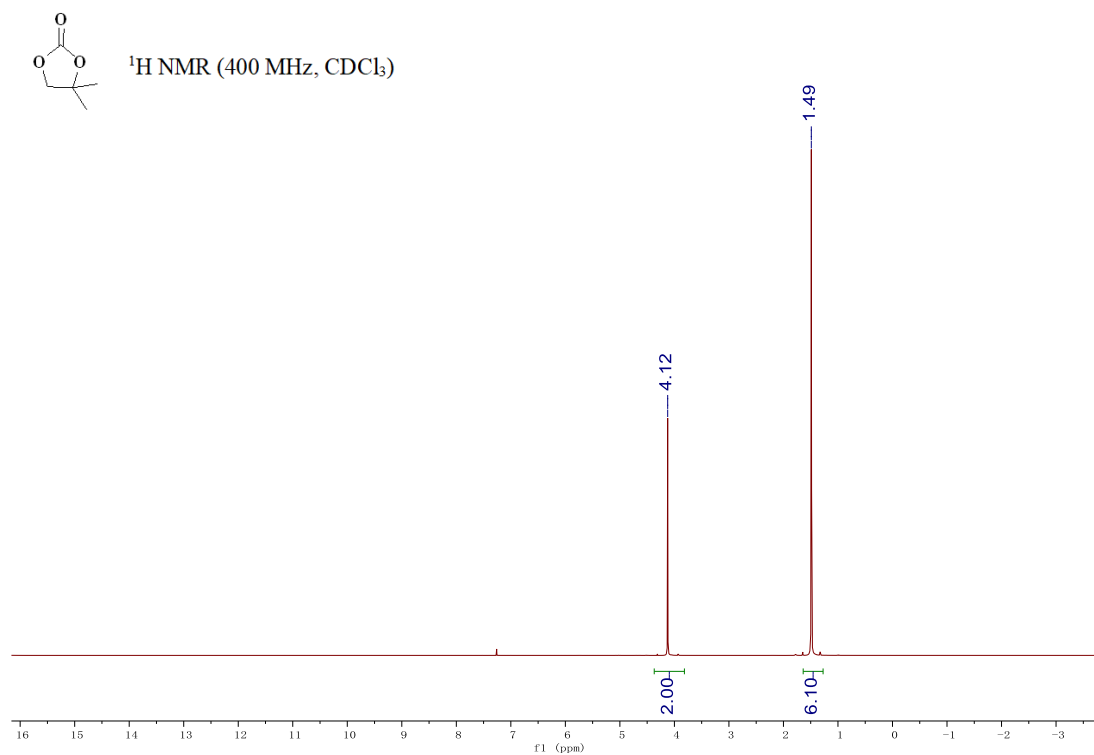


Figure S30. ^1H NMR Spectra of 4,4-Dimethyl-1,3-dioxolan-2-one (2k).

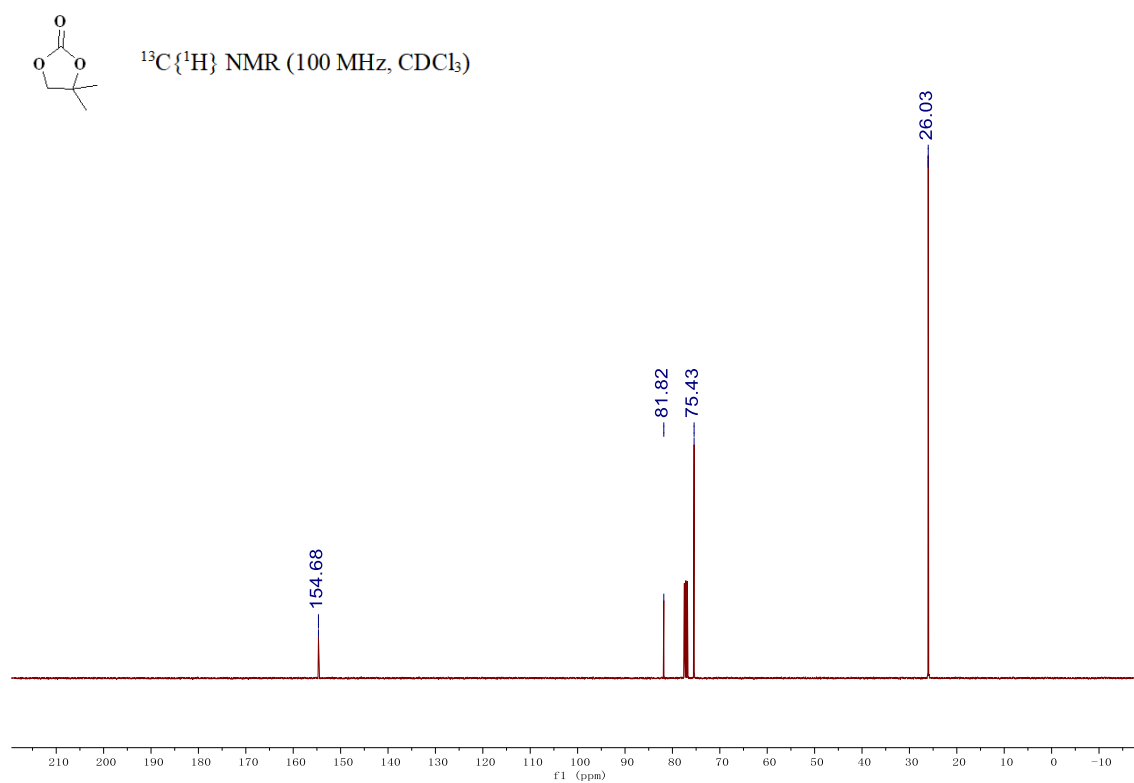


Figure S31. $^{13}\text{C}\{^1\text{H}\}$ NMR Spectra of 4,4-Dimethyl-1,3-dioxolan-2-one (2k).

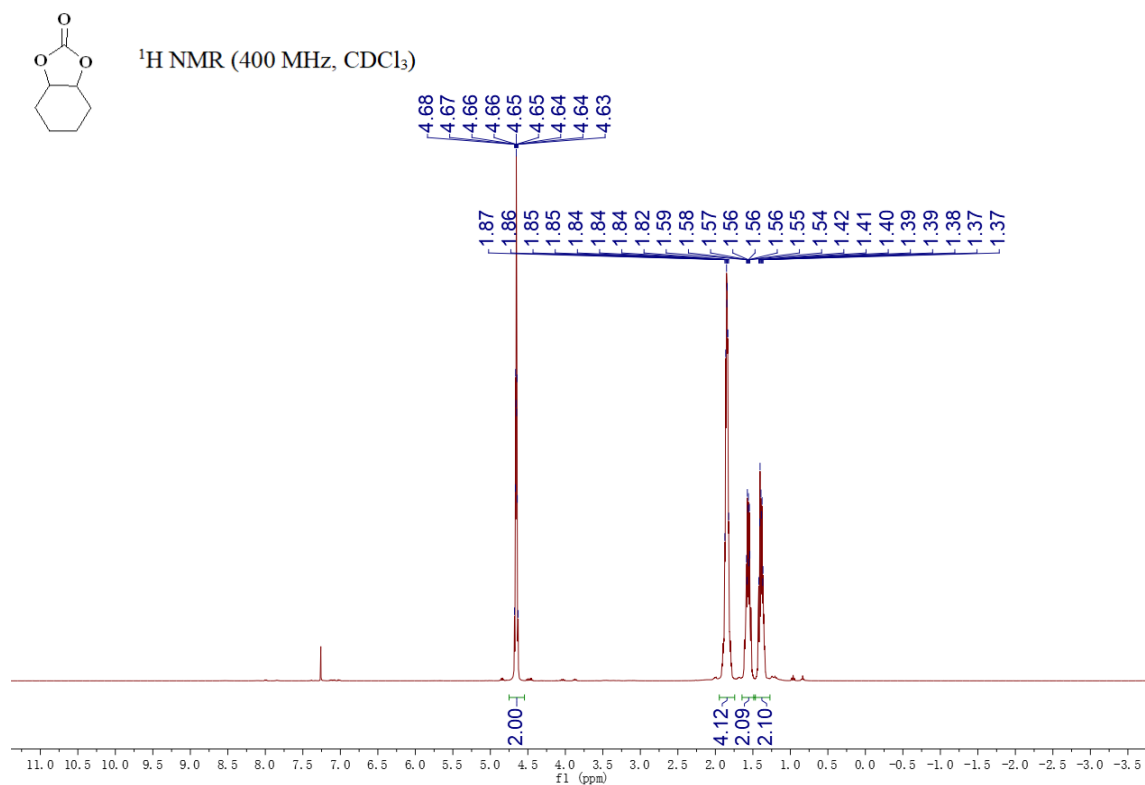


Figure S32. ¹H NMR Spectra of Hexahydrobenzo[d][1,3]dioxol-2-one (21).

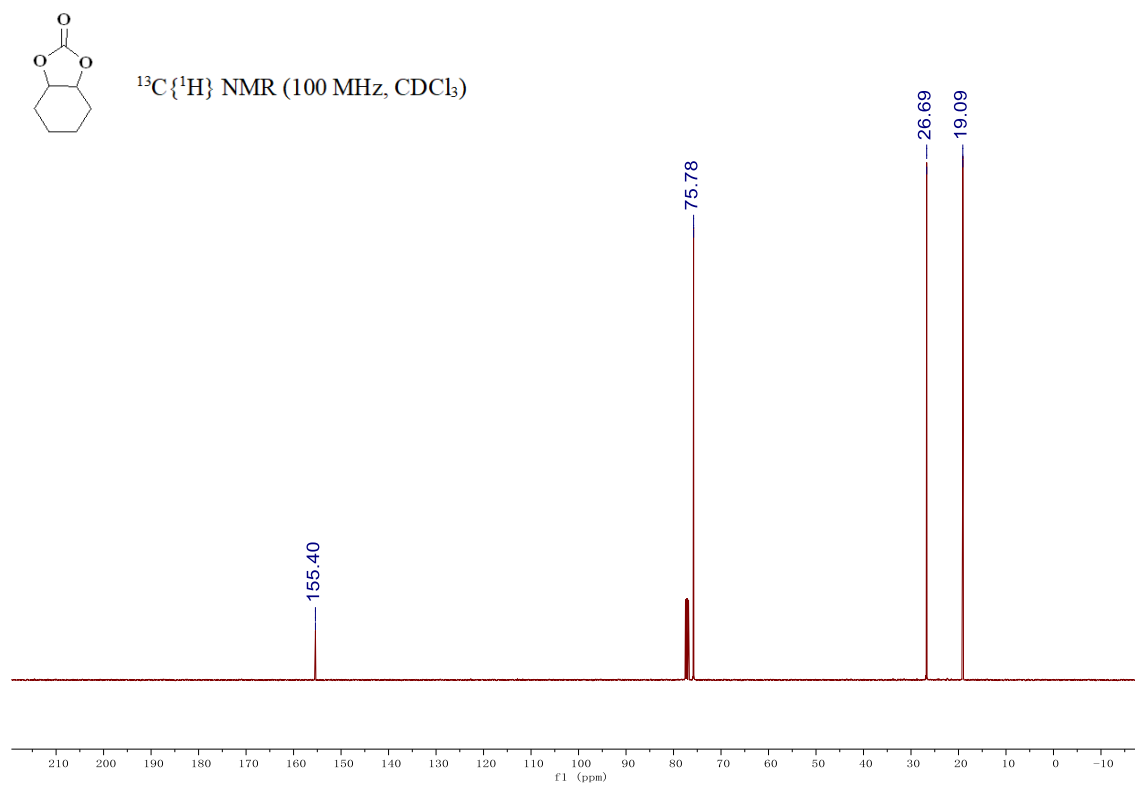


Figure S33. ¹³C{¹H} NMR Spectra of Hexahydrobenzo[d][1,3]dioxol-2-one (21).

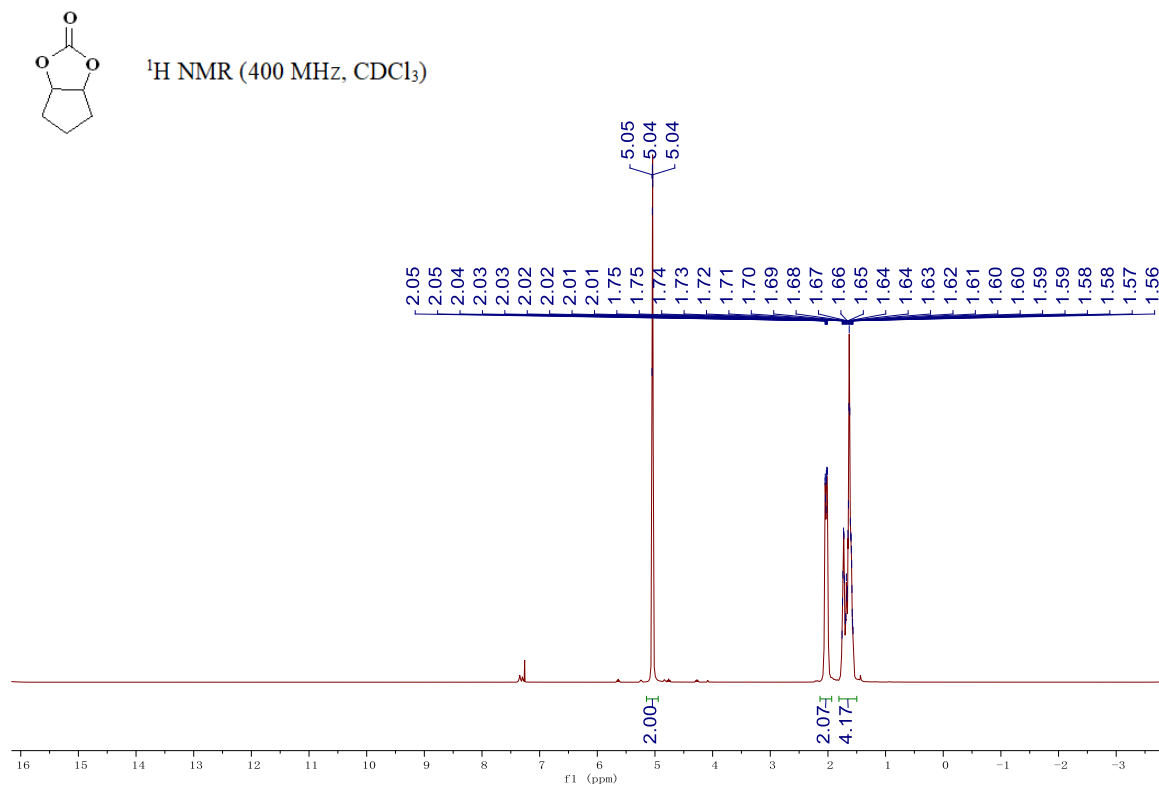


Figure S34. ¹H NMR Spectra of (3aR,6aS)-Tetrahydro-4H-cyclopenta[d][1,3]dioxol-2-one (2m).

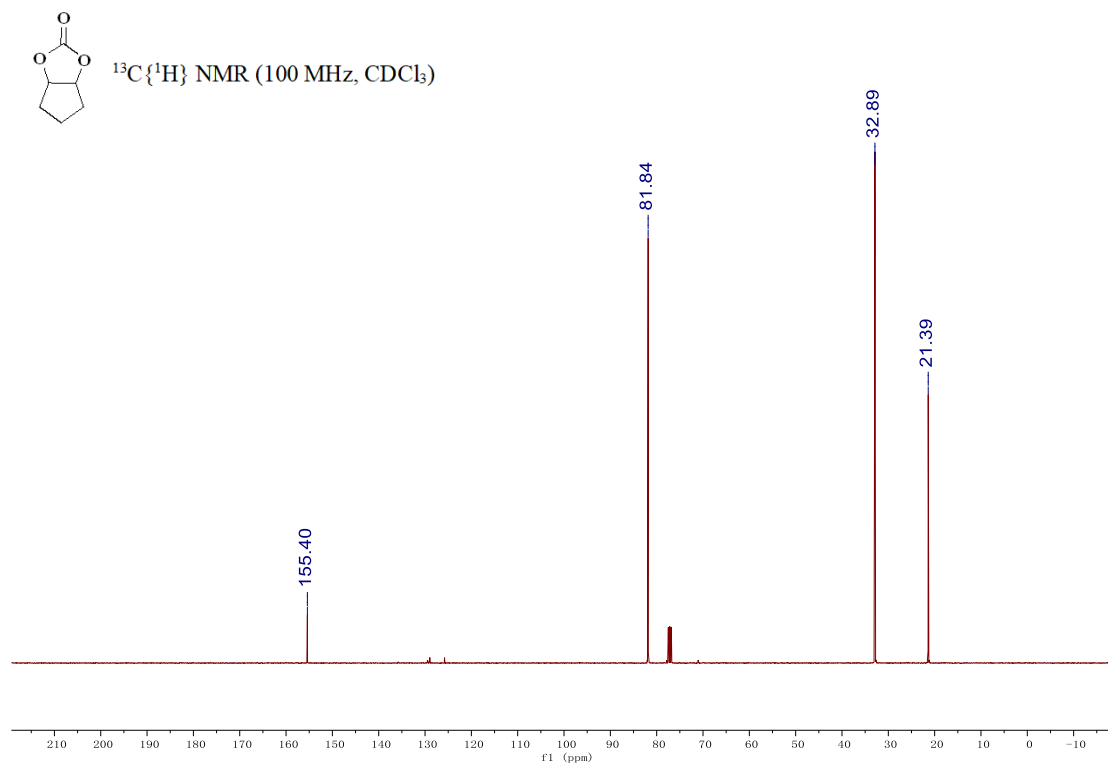


Figure S35. ¹³C{¹H} NMR Spectra of (3aR,6aS)-Tetrahydro-4H-cyclopenta[d][1,3]dioxol-2-one (2m).

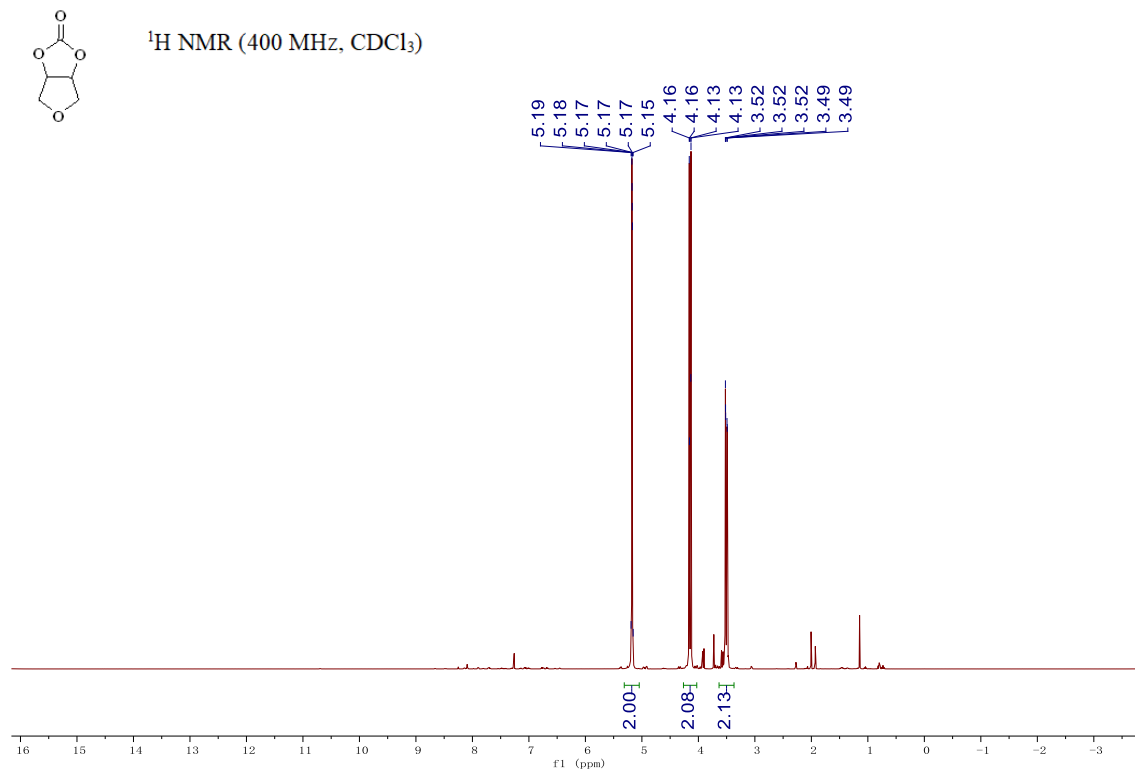


Figure S36. ^1H NMR Spectra of (3aR,6aS)-Tetrahydrofuro[3,4-d][1,3]dioxol-2-one (2n).

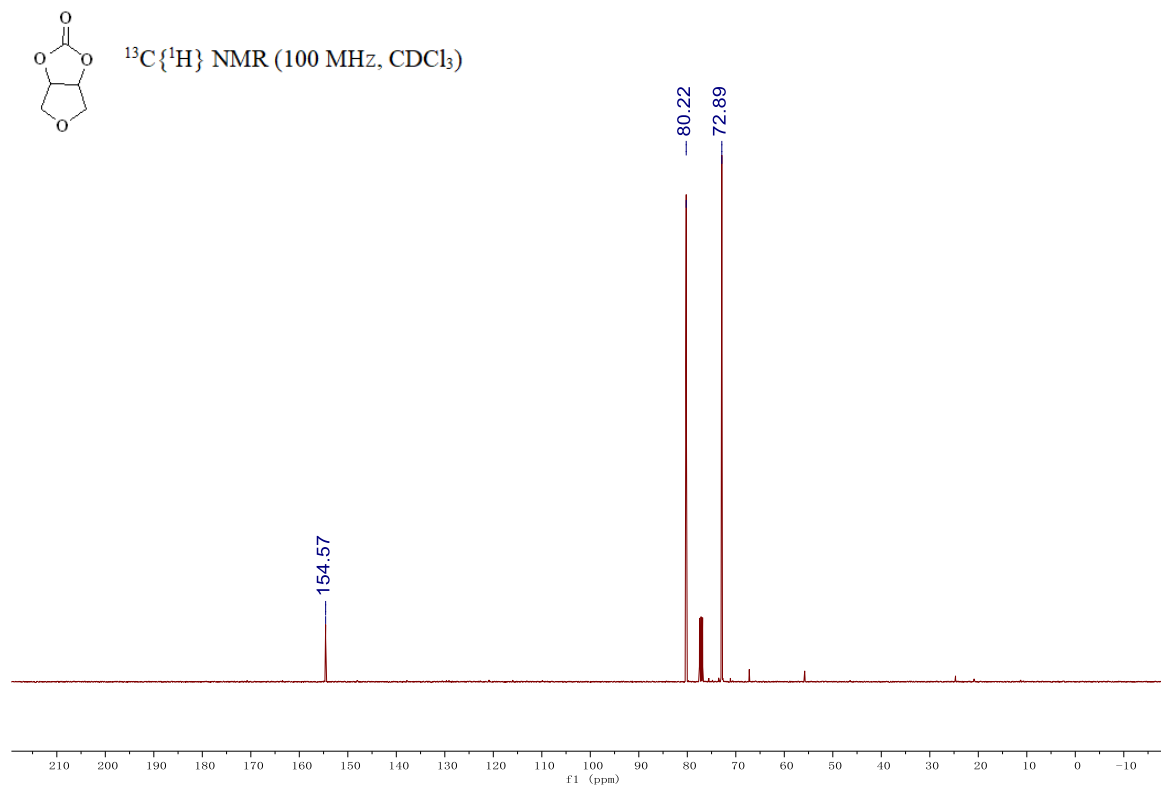


Figure S37. $^{13}\text{C}\{^1\text{H}\}$ NMR Spectra of (3aR,6aS)-Tetrahydrofuro[3,4-d][1,3]dioxol-2-one (2n).

8. References

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