Electronic Supplementary Material (ESI) for RSC Advances.

Supporting Information

CH_2Cl_2 as reagent in synthesis of methylene-bridged 3,3′-bis(oxazolidin-2-one) derivatives under ambient conditions

Qingfeng Liu, Yansen Zhang, Zhiguo Zhang, Tongxin Liu, Lei Shi, Guisheng Zhang*

Collaborative Innovation Center of Henan Province for Green Manufacturing of Fine Chemicals, Key Laboratory of Green Chemical Media and Reactions, Ministry of Education, School of Chemistry and Chemical Engineering, Henan Normal University, Xinxiang, Henan 453007, P. R. China

Fax: (+86) 373-3325250 E-mail: zgs6668@yahoo.com

Table of Contents

I. *X*-ray single crystal diffraction data of **2a**

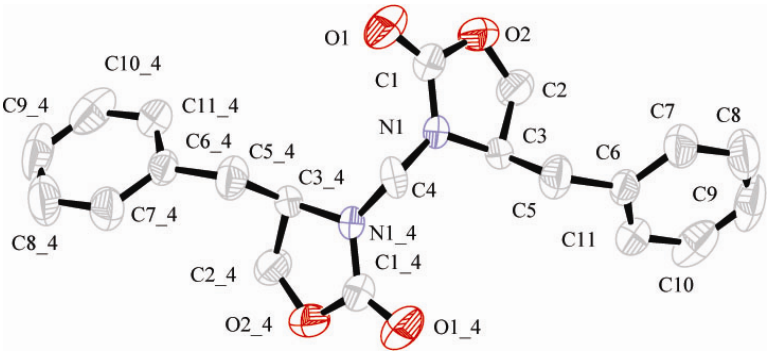


Table 1. Crystal data and structure refinement for **2a**

Identification code	**2a**
Empirical formula	$C_{21}H_{22}N_2O_4$
Formula weight	366.41
Temperature	293(2) K
Wavelength	0.7107 A
Crystal system, space group	Monoclinic, P 21/c
Unit cell dimensions	a = 10.0068(4) A alpha = 90.00 deg.
	b = 10.0068(4) A beta = 90.00 deg.
	c = 16.3846(11) A gamma =120.00 deg.
Volume	1420.88(12) A^3
Z, Calculated density	21, 1.365 Mg/m^3
Absorption coefficient	0.090 mm^-1
F(000)	582
Theta range for data collection	1.17 to 35.15 deg.
Limiting indices	-11<=h<=12, -12<=k<=11, -11<=l<=19
Reflections collected / unique	7548 / 2011 [R(int) = 0.0261]
Completeness to theta = 25.00	99.8 %
Absorption correction	Empirical
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2011 / 0 / 124
Goodness-of-fit on F^2	1.014
Final R indices [I>2sigma(I)]	R1 = 0.0528, wR2 = 0.1003
R indices (all data)	R1 = 0.0888, wR2 = 0.1154
Largest diff. peak and hole	0.175 and -0.247 e.A^-3

Table 2. Bond lengths [A] and angles [deg] for **2a**

C(1)-O(1)	1.212(3)	O(2)-C(2)-H(2A)	110.5
C(1)-N(1)	1.339(3)	C(3)-C(2)-H(2A)	110.5
C(1)-O(2)	1.341(4)	O(2)-C(2)-H(2B)	110.5
C(2)-O(2)	1.430(4)	C(3)-C(2)-H(2A)	110.5
C(2)-C(3)	1.513(4)	H(2A)-C(2)-H(2B)	108.7
C(2)-H(2A)	0.9700	N(1)-C(3)-C(2)	99.3(2)
C(2)-H(2B)	0.9700	N(1)-C(3)-C(5)	111.4(2)
C(3)-N(1)	1.457(3)	C(2)-C(3)-C(5)	114.5(2)
C(3)-H(3)	0.9800	N(1)-C(3)-H(3)	110.4
C(4)-N(1)	1.440(3)	C(2)-C(3)-H(3)	110.4
C(4)-H(4A)	0.9700	C(5)-C(3)-H(3)	110.4
C(4)-H(4B)	0.9700	N(1)-C(4)-N(1)	112.4(4)
C(5)-C(6)	1.505(4)	N(1)-C(4)-H(4A)	109.1
C(5)-H(5A)	0.9700	N(1)-C(4)-H(4B)	109.1
C(5)-H(5B)	0.9700	H(4A)-C(4)-H(4B)	107.9
C(6)-C(7)	1.373(4)	C(6)-C(5)-C(3)	113.9(2)
C(6)-C(11)	1.375(4)	C(6)-C(5)-H(5A)	108.8
C(7)-C(8)	1.357(5)	C(3)-C(5)-H(5A)	108.8
C(7)-H(7)	0.9300	C(6)-C(5)-H(5B)	108.8
C(8)-C(9)	1.342(6)	C(3)-C(5)-H(5B)	108.8
C(8)-H(8)	0.9300	H(5A)-C(5)-H(5B)	107.7
C(9)-C(10)	1.384(6)	C(7)-C(6)-C(11)	118.4(3)
C(9)-H(9)	0.9300	C(7)-C(6)-C(5)	119.6(3)
C(10)-C(11)	1.387(5)	C(11)-C(6)-C(5)	122.0(3)
C(10)-H(10)	0.9300	C(8)-C(7)-C(6)	121.7(4)
C(11)-H(11)	0.9300	C(8)-C(7)-H(7)	119.2
		C(6)-C(7)-H(7)	119.2
O(1)-C(1)-N(1)	127.5(3)	C(9)-C(8)-C(7)	120.4(4)

O(1)-C(1)-O(2)	122.4(3)	C(9)-C(8)-H(8)	119.8
N(1)-C(1)-O(2)	110.1(3)	C(7)-C(8)-H(8)	119.8
O(2)-C(2)-C(3)	106.2(2)	C(8)-C(9)-C(10)	120.0(4)
C(8)-C(9)-H(9)	120.0	C(2)-C(3)-N(1)-C(1)	-16.6(3)
C(10)-C(9)-H(9)	120.0	C(5)-C(3)-N(1)-C(1)	104.3(3)
C(9)-C(10)-C(11)	119.6(4)	C(2)-C(3)-N(1)-C(4)	178.4(3)
C(9)-C(10)-H(10)	120.2	C(5)-C(3)-N(1)-C(4)	-60.6(3)
C(11)-C(10)-H(10)	120.2	O(1)-C(1)-O(2)-C(2)	-175.0(3)
C(6)-C(11)-C(10)	119.9(3)	N(1)-C(1)-O(2)-C(2)	6.0(4)
C(6)-C(11)-H(11)	120.0	C(3)-C(2)-O(2)-C(1)	-16.5(3)
C(10)-C(11)-H(11)	120.0		
C(1)-N(1)-C(4)	122.1(2)		
C(1)-N(1)-C(3)	111.9(2)		
C(4)-N(1)-C(3)	124.19(17)		
C(1)-O(2)-C(2)	108.6(2)		

O(2)-C(2)-C(3)-N(1)	19.1(3)
O(2)-C(2)-C(3)-C(5)	-99.5(3)
N(1)-C(3)-C(5)-C(6)	-170.0(2)
C(2)-C(3)-C(5)-C(6)	-58.4(4)
C(3)-C(5)-C(6)-C(7)	115.9(3)
C(3)-C(5)-C(6)-C(11)	-64.9(4)
C(11)-C(6)-C(7)-C(8)	-0.7(5)
C(5)-C(6)-C(7)-C(8)	178.6(3)
C(6)-C(7)-C(8)-C(9)	1.3(6)
C(7)-C(8)-C(9)-C(10)	-0.9(6)
C(8)-C(9)-C(10)-C(11)	0.0(5)
C(7)-C(6)-C(11)-C(10)	-0.3(4)
C(5)-C(6)-C(11)-C(10)	-179.5(3)
C(9)-C(10)-C(11)-C(6)	0.6(5)

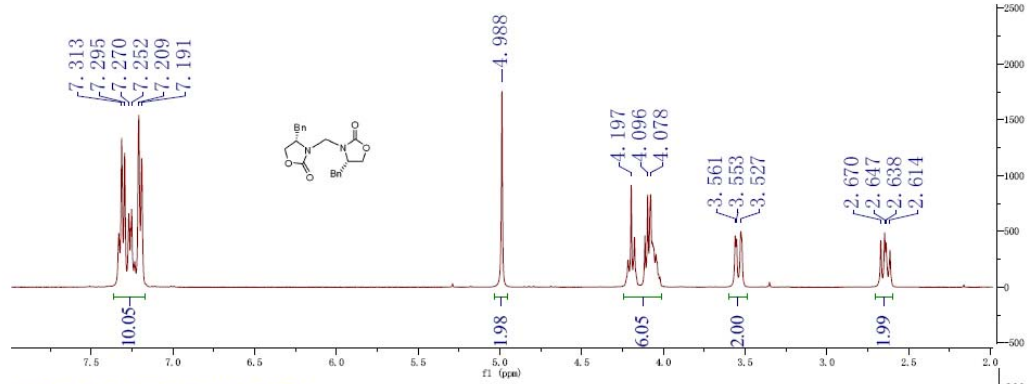
O(1)-C(1)-N(1)-C(4)	-5.9(5)
O(2)-C(1)-N(1)-C(4)	173.0(3)
O(1)-C(1)-N(1)-C(3)	-171.3(3)
O(2)-C(1)-N(1)-C(3)	7.7(4)
N(1)-C(4)-N(1)-C(1)	117.4(3)
N(1)-C(4)-N(1)-C(3)	-79.1(2)

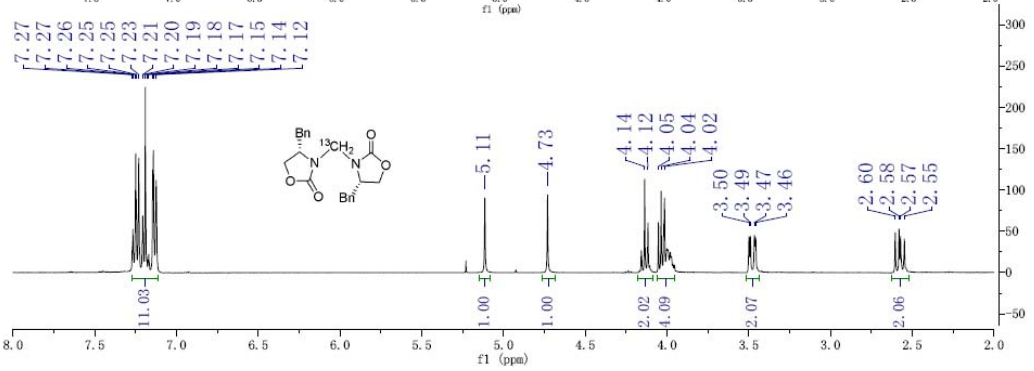
II. For the $^{13}CH_2Cl_2$ Labeled Experiments

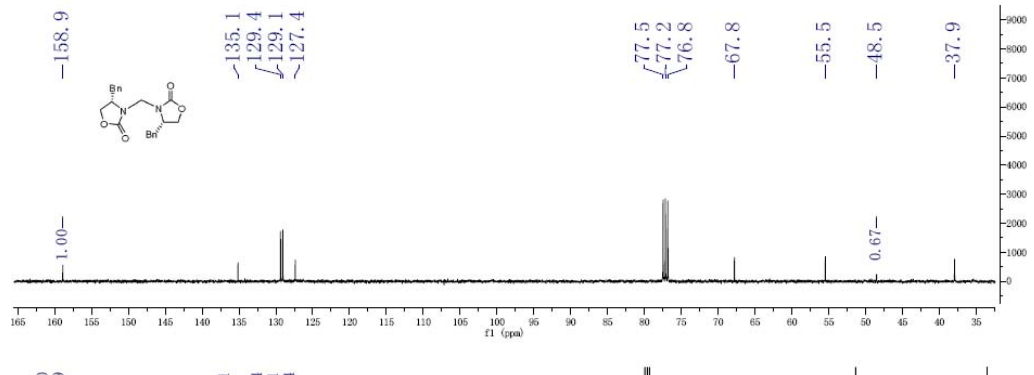
The $^{13}CH_2Cl_2$ was purchased from Cambridge Isotope Laboratories, Inc. ($^{13}CH_2Cl_2$ (^{13}C, 99%)) and used without further purification.

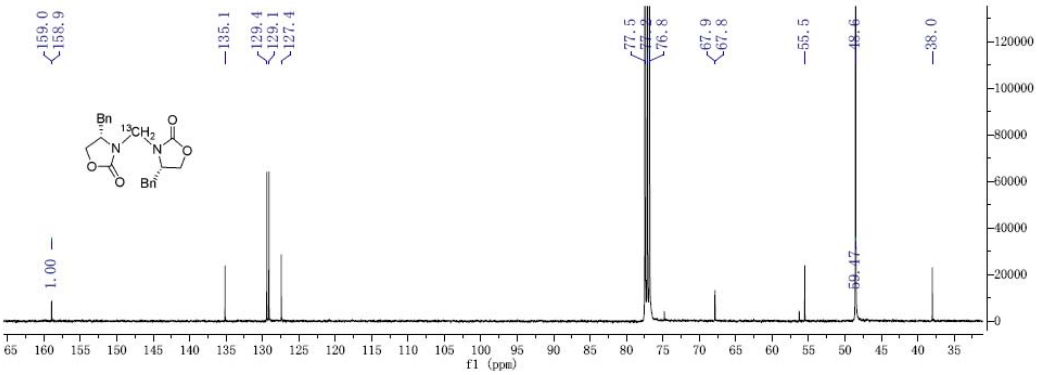
^{13}C Enrichments of afforded were calculated from the integrates resonances detected by inverse gated decoupling ^{13}C NMR. Carbon resonances were integrated and standardized to three different positions (C (159.0 ppm), C (135.1 ppm), and C (127.4 ppm)). The ratio of the integrated signal from the labeled sample to unlabeled product was determined, then the three sets were averaged. This quotient was multiplied by the natural abundance of ^{13}C (1.1%) to give the percent ^{13}C incorporations at each carbon position of ***^{13}C*-2a**.

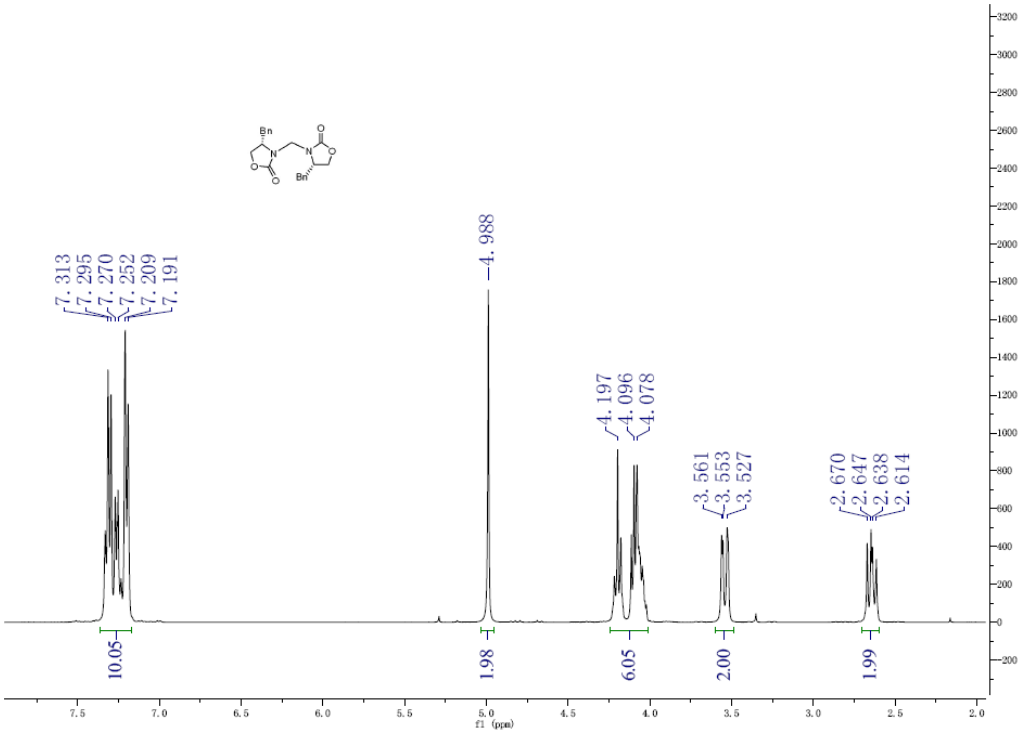
	Carbon	-CH_2-
	Authentic δ (ppm)	48.5
	Labeled δ (ppm)	48.6
C (159.0 ppm)	Ratio Labeled	59.47
	Ratio Unlabeled	0.67
	Labeled/Unlabeled	88.76
C (135.1 ppm)	Ratio Labeled	52.63
	Ratio Unlabeled	0.58
	Labeled/Unlabeled	90.74
C (127.4 ppm)	Ratio Labeled	41.71
	Ratio Unlabeled	0.46
	Labeled/Unlabeled	90.67
Average Labeled/Unlabeled		90.06
Absolute Abundance (%)		99.06

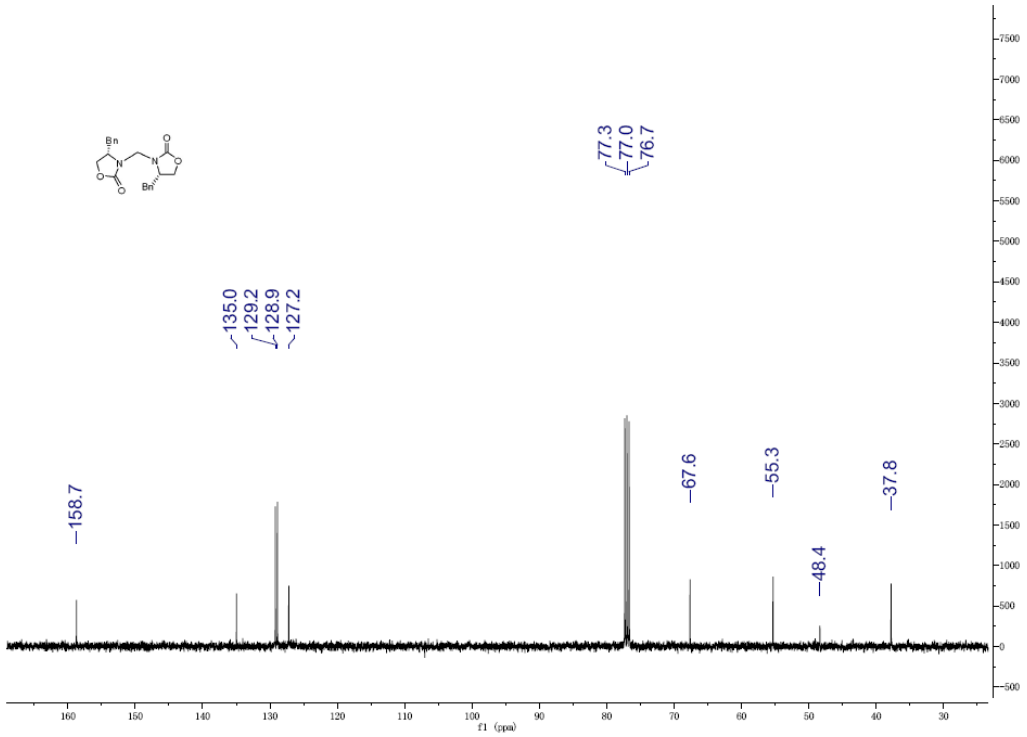

7.313
7.295
7.270
7.252
7.209
7.191
4.988
4.197
4.096
4.078
3.561
3.553
3.527
2.670
2.647
2.638
2.614
10.05
1.98
6.05
2.00
1.99
f1 (ppm)

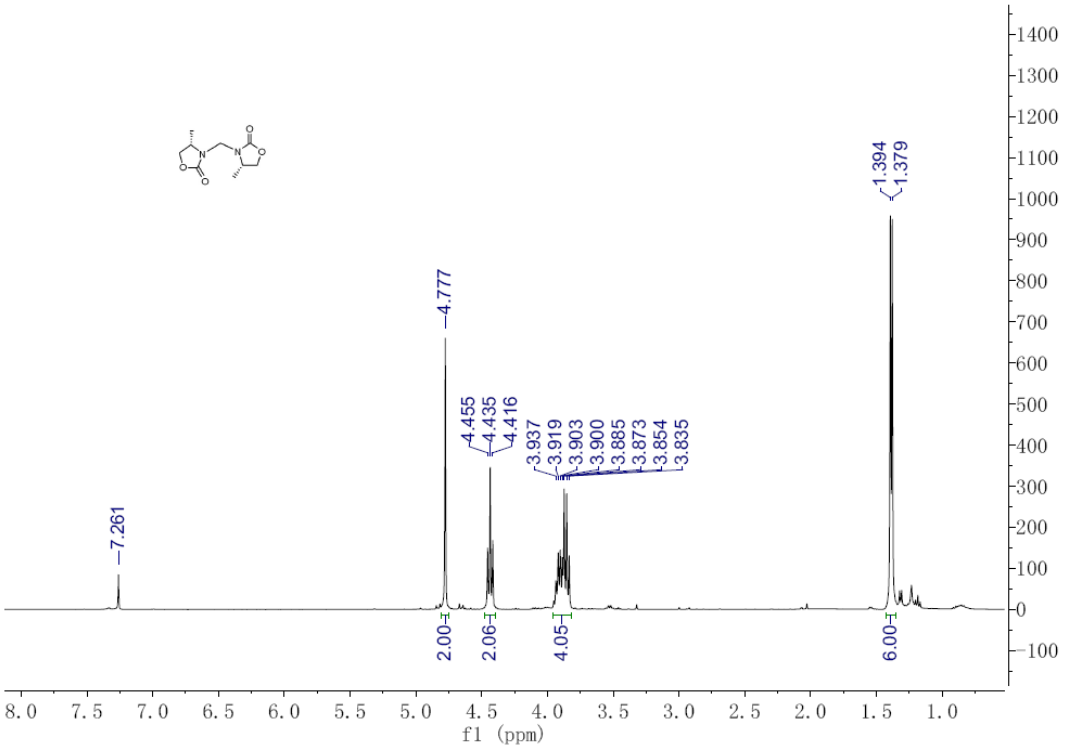

7.27
7.27
7.26
7.25
7.25
7.23
7.21
7.20
7.19
7.18
7.17
7.15
7.14
7.12
5.11
4.73
4.14
4.12
4.05
4.04
4.02
3.50
3.49
3.47
3.46
2.60
2.58
2.57
2.55
11.03
1.00
1.00
2.02
4.09
2.07
2.06
f1 (ppm)

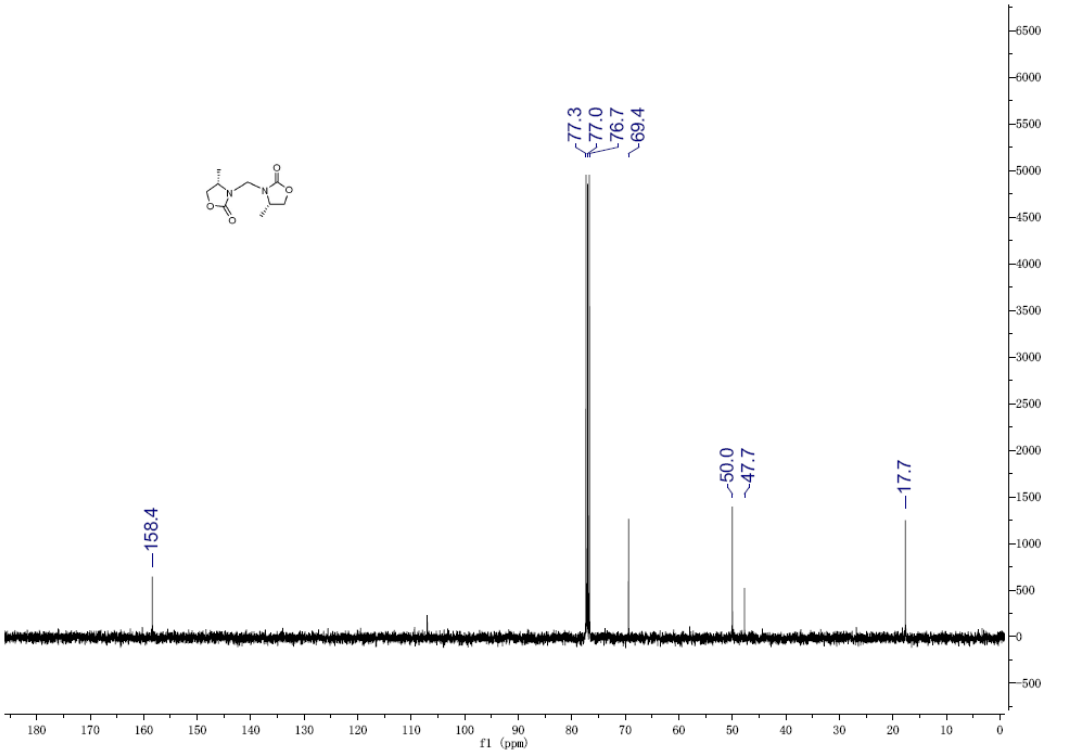

158.9
135.1
129.4
129.1
127.4
77.5
77.2
76.8
67.8
55.5
48.5
37.9
1.00
0.67
f1 (ppm)

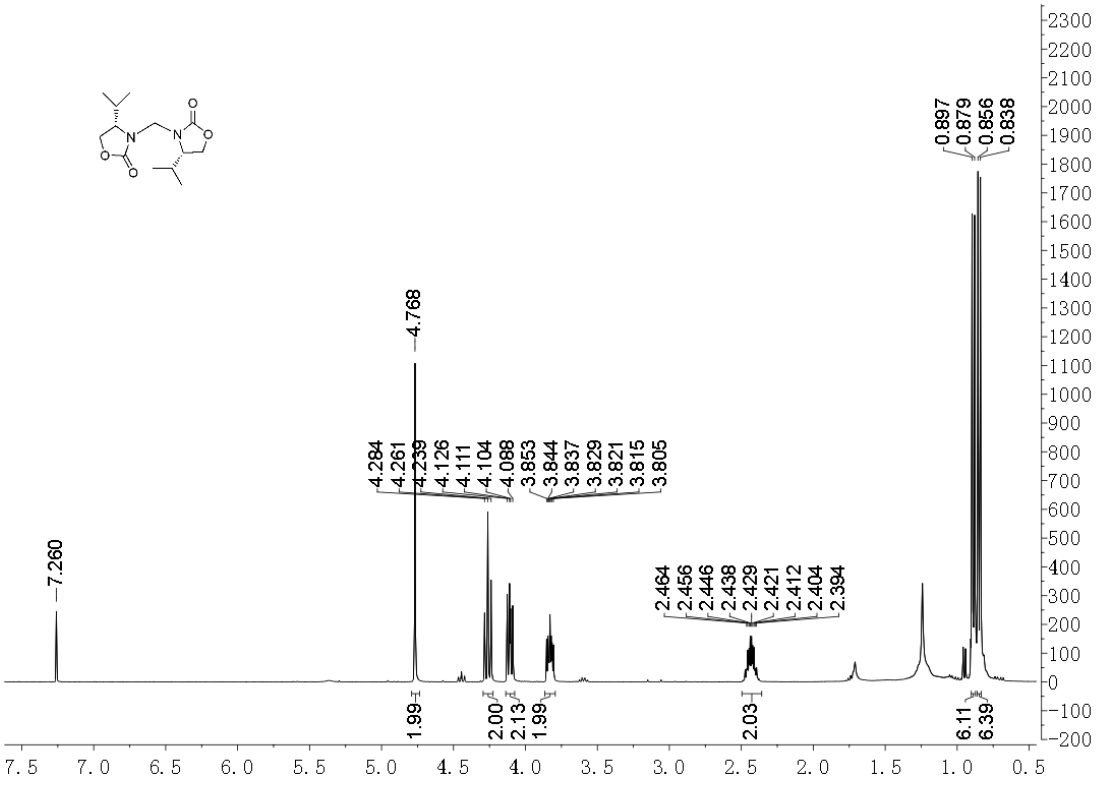

159.0
158.9
135.1
129.4
129.1
127.4
77.5
76.8
67.9
67.8
55.5
48.6
38.0
1.00
68.47
f1 (ppm)

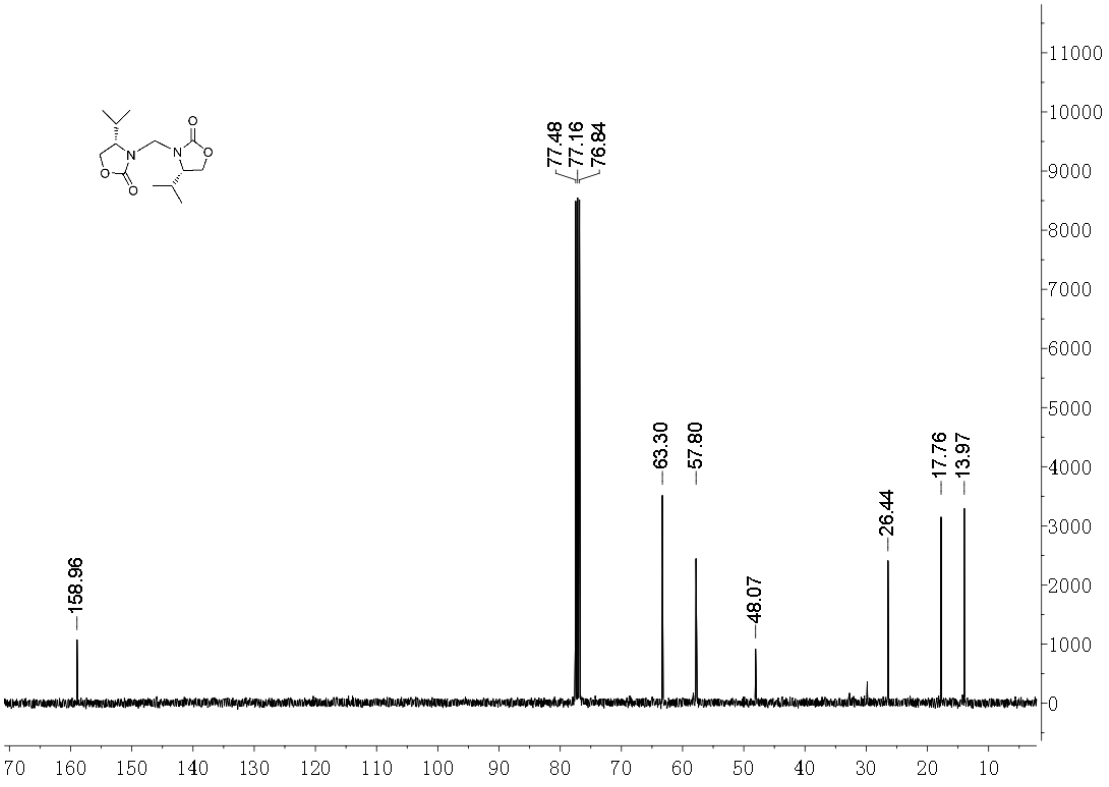

7.313
7.295
7.270
7.252
7.209
7.191
4.988
4.197
4.096
4.078
3.561
3.553
3.527
2.670
2.647
2.638
2.614
10.05
1.98
6.05
2.00
1.99
f1 (ppm)

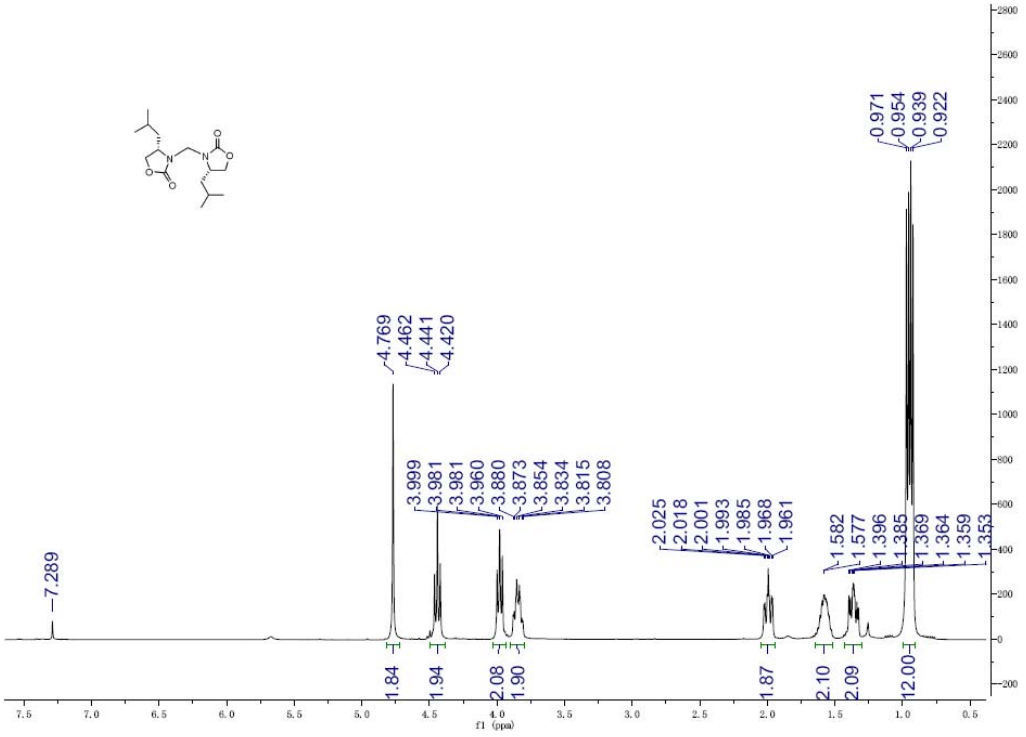

77.3
77.0
76.7
135.0
129.2
128.9
127.2
158.7
67.6
55.3
48.4
37.8
f1 (ppm)

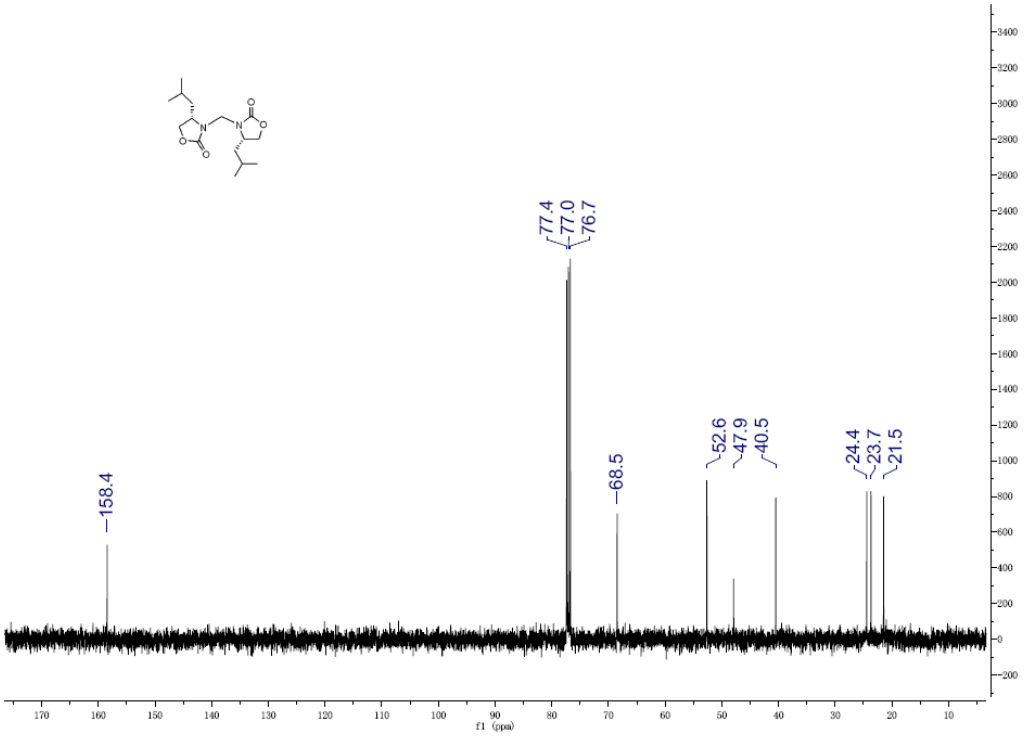

7.261
4.777
4.455
4.435
4.416
3.937
3.919
3.903
3.900
3.885
3.873
3.854
3.835
1.394
1.379
2.00
2.06
4.05
6.00
f1 (ppm)

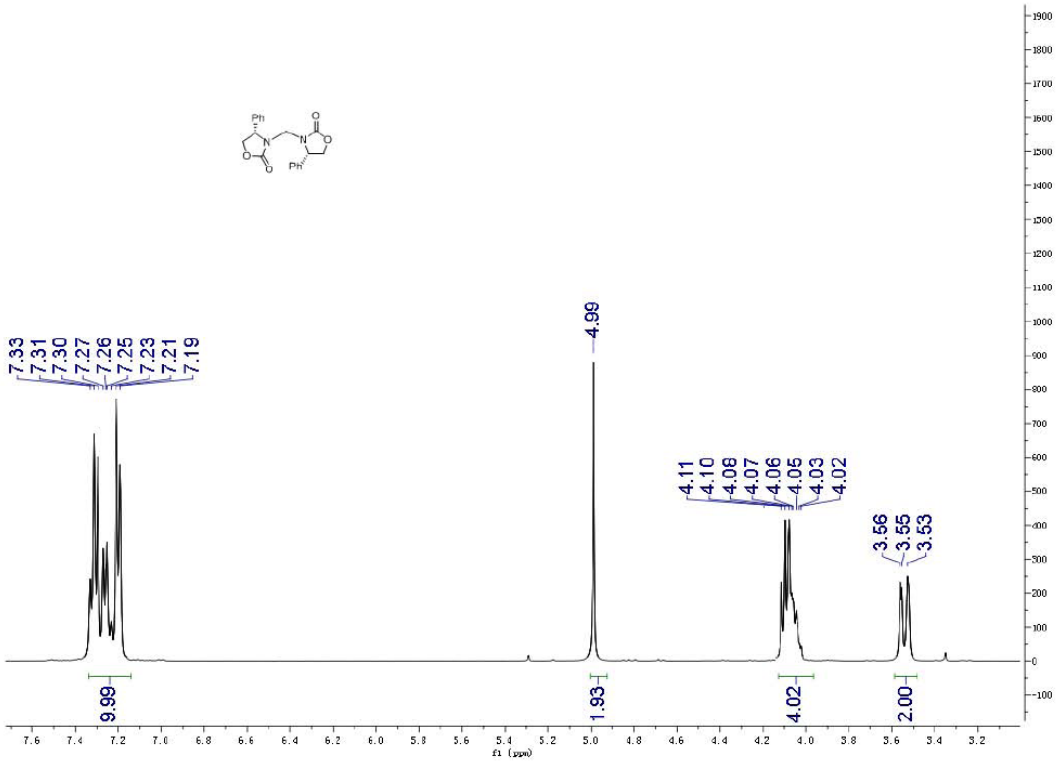

158.4
77.3
77.0
76.7
69.4
50.0
47.7
17.7
f1 (ppm)

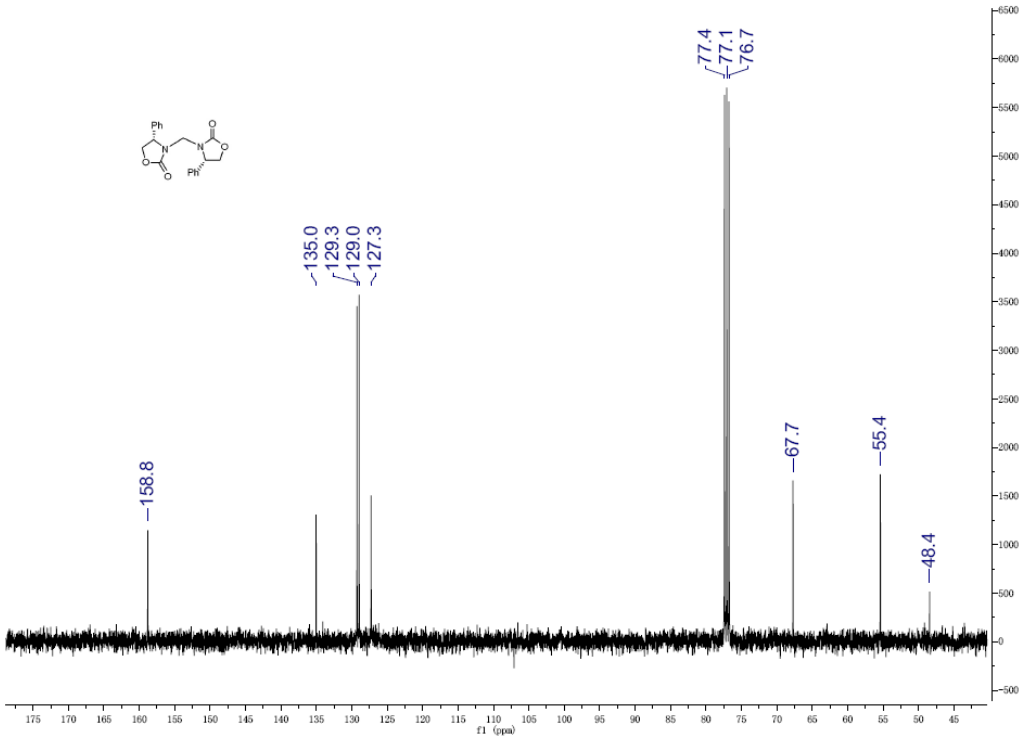

7.260
4.768
4.284
4.261
4.239
4.126
4.111
4.104
4.088
3.853
3.844
3.837
3.829
3.821
3.815
3.805
2.464
2.456
2.446
2.438
2.429
2.421
2.412
2.404
2.394
0.897
0.879
0.856
0.838
1.99
2.00
2.13
1.99
2.03
6.11
6.39

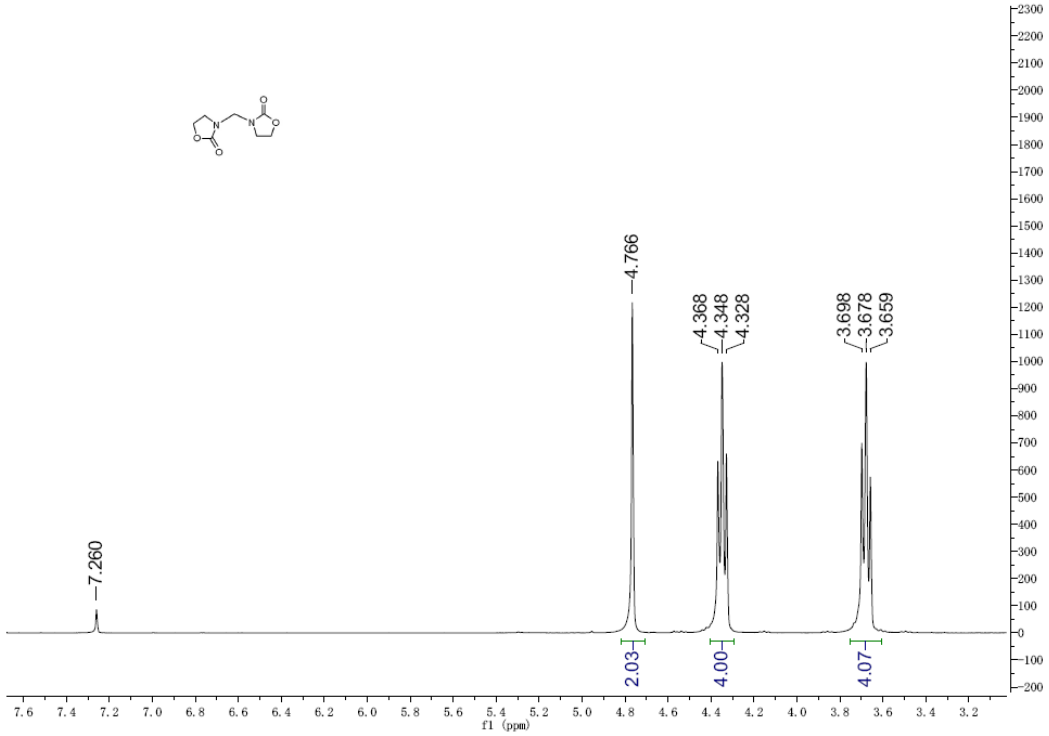

158.96
77.48
77.16
76.84
63.30
57.80
48.07
26.44
17.76
13.97

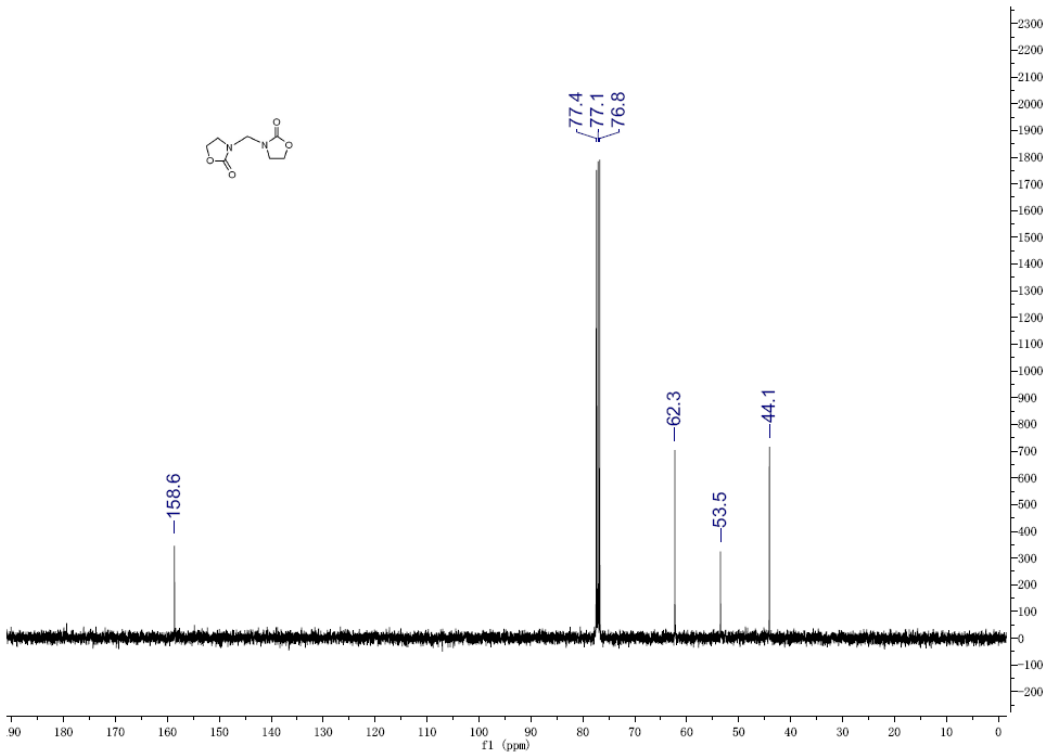

7.289
4.769
4.462
4.441
4.420
3.999
3.981
3.981
3.960
3.880
3.873
3.854
3.834
3.815
3.808
2.025
2.018
2.001
1.993
1.985
1.968
1.961
1.582
1.577
1.396
1.385
1.369
1.364
1.359
1.353
0.971
0.954
0.939
0.922
1.84
1.94
2.08
1.90
1.87
2.10
2.09
12.00
f1 (ppm)

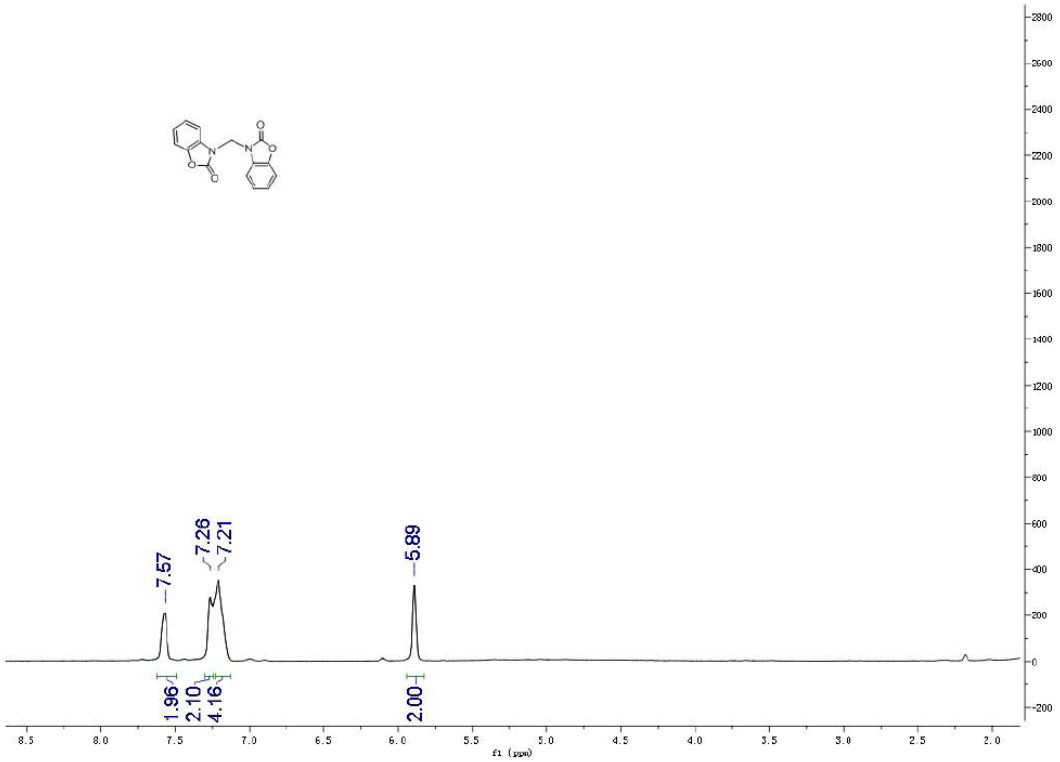

158.4
77.4
77.0
76.7
68.5
52.6
47.9
40.5
24.4
23.7
21.5
f1 (ppm)

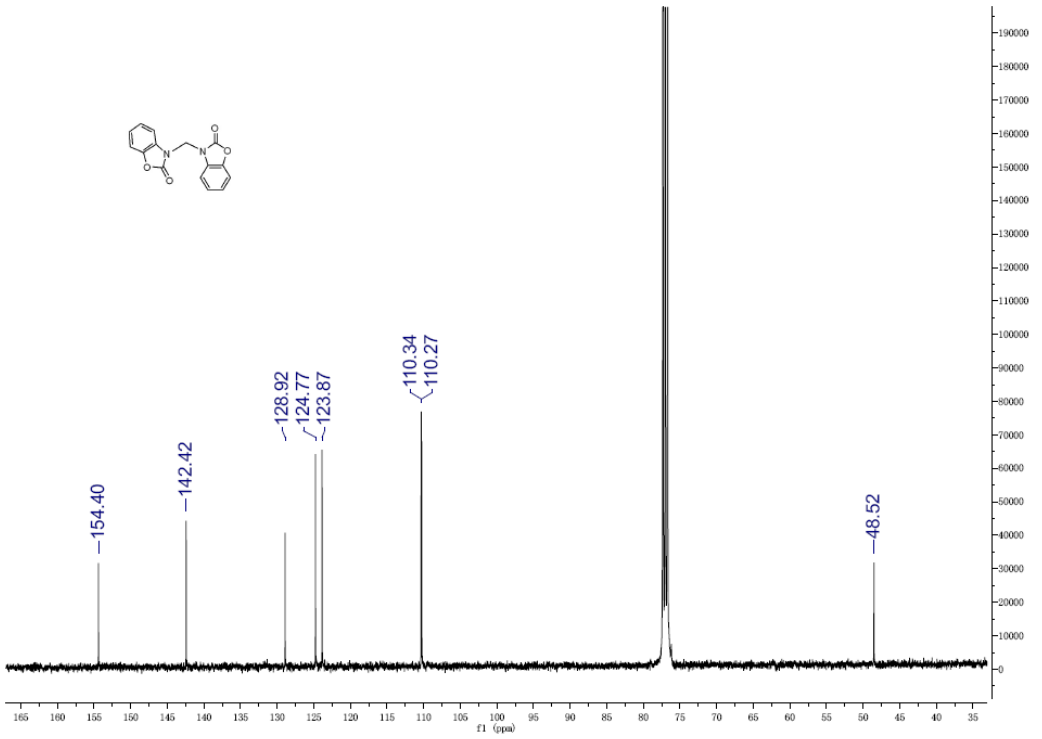

7.33
7.31
7.30
7.27
7.26
7.25
7.23
7.21
7.19
4.99
4.11
4.10
4.08
4.07
4.06
4.05
4.03
4.02
3.56
3.55
3.53
9.99
1.93
4.02
2.00
f1 (ppm)

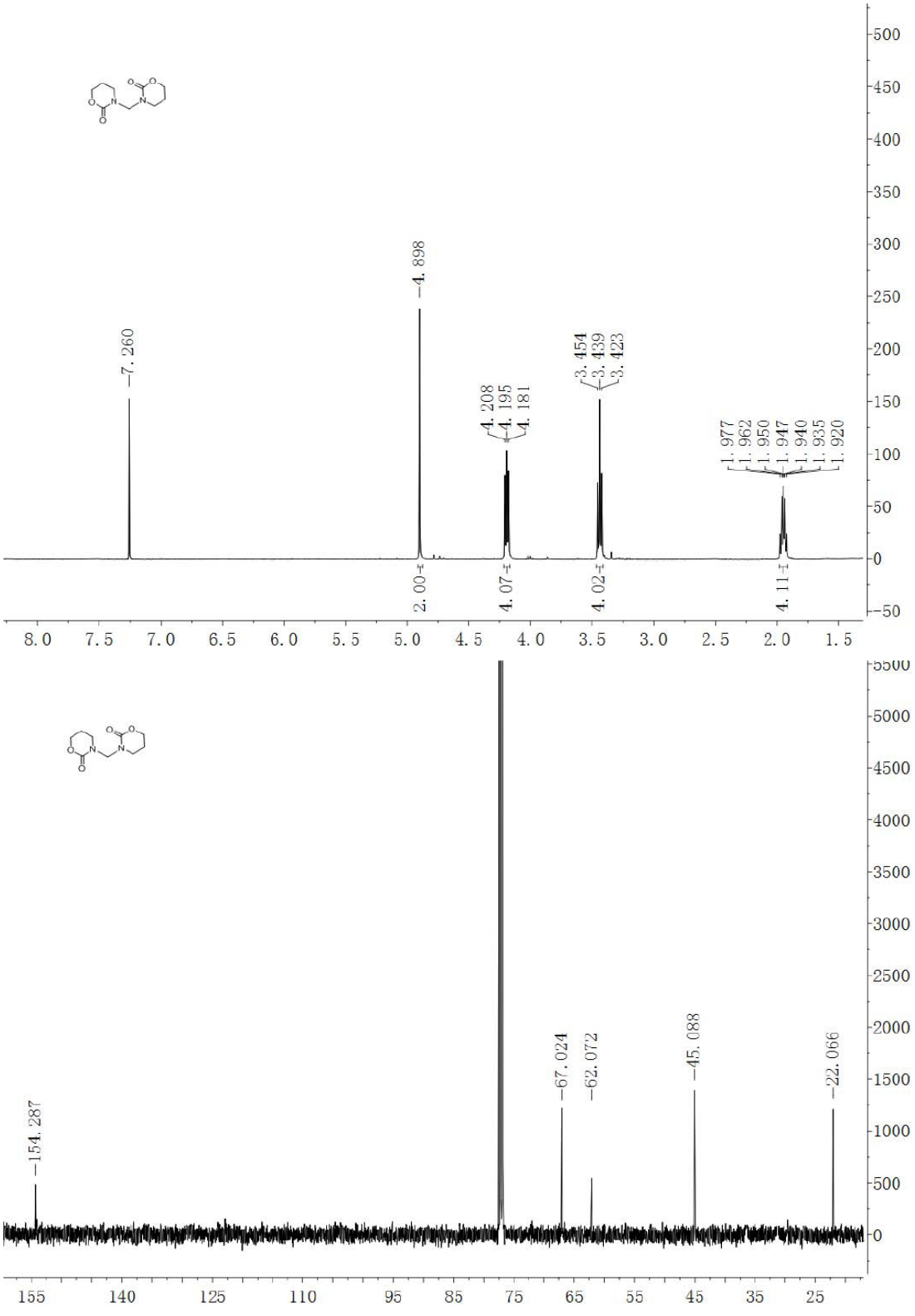

77.4
77.1
76.7
135.0
129.3
129.0
127.3
158.8
67.7
55.4
48.4
f1 (ppm)

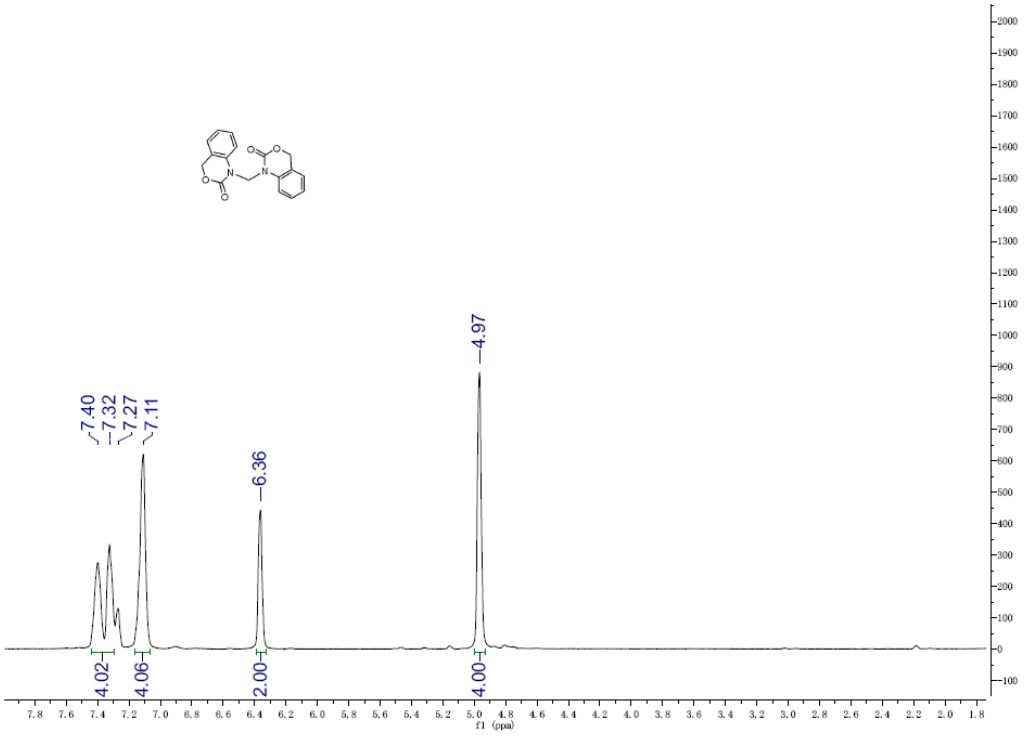

7.260
4.766
4.368
4.348
4.328
3.698
3.678
3.659
2.03
4.00
4.07
f1 (ppm)

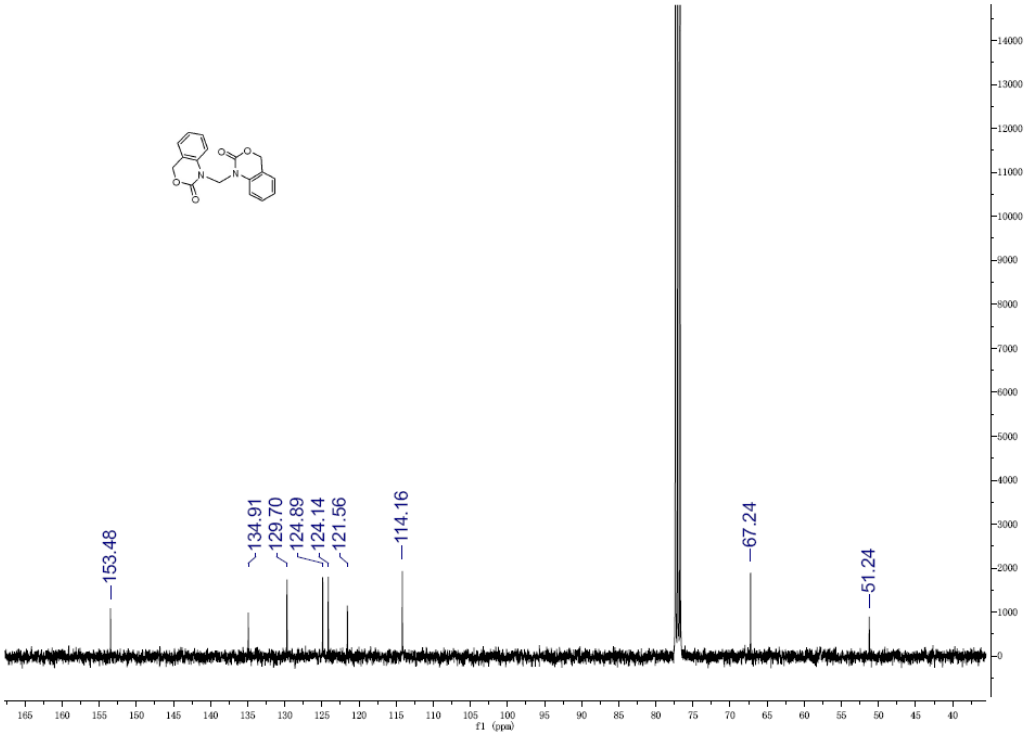

158.6
77.4
77.1
76.8
62.3
53.5
44.1
f1 (ppm)

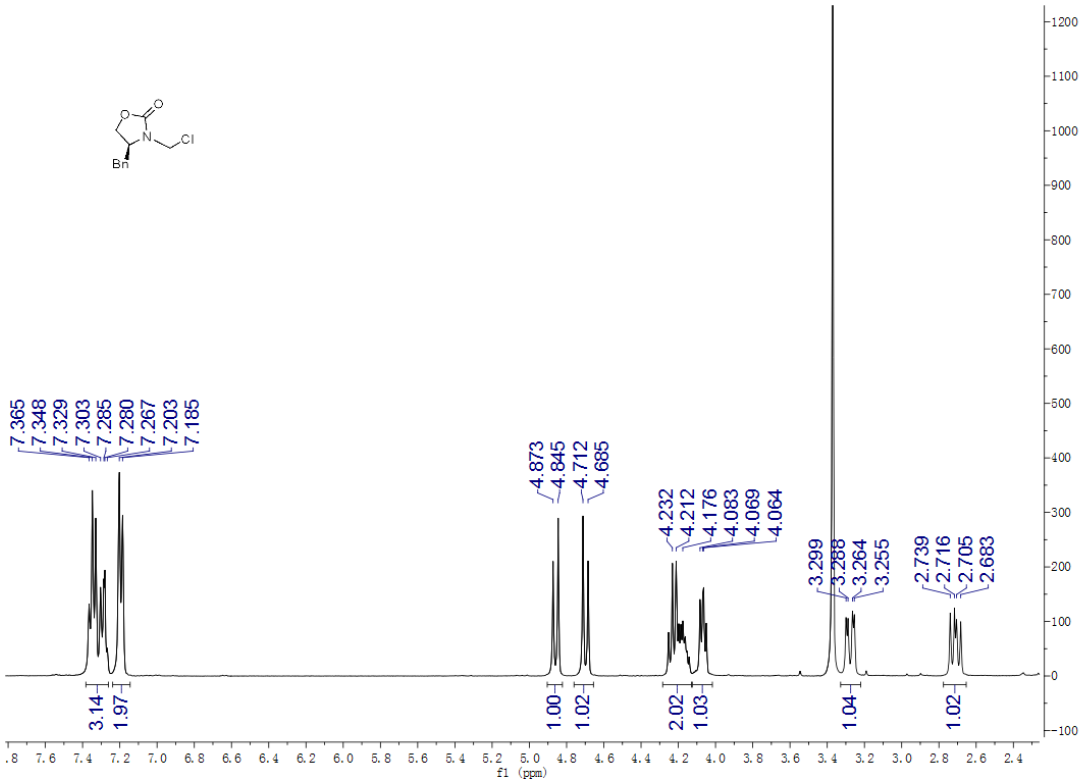

7.57
7.26
7.21
5.89
1.96
2.10
4.16
2.00
f1 (ppm)

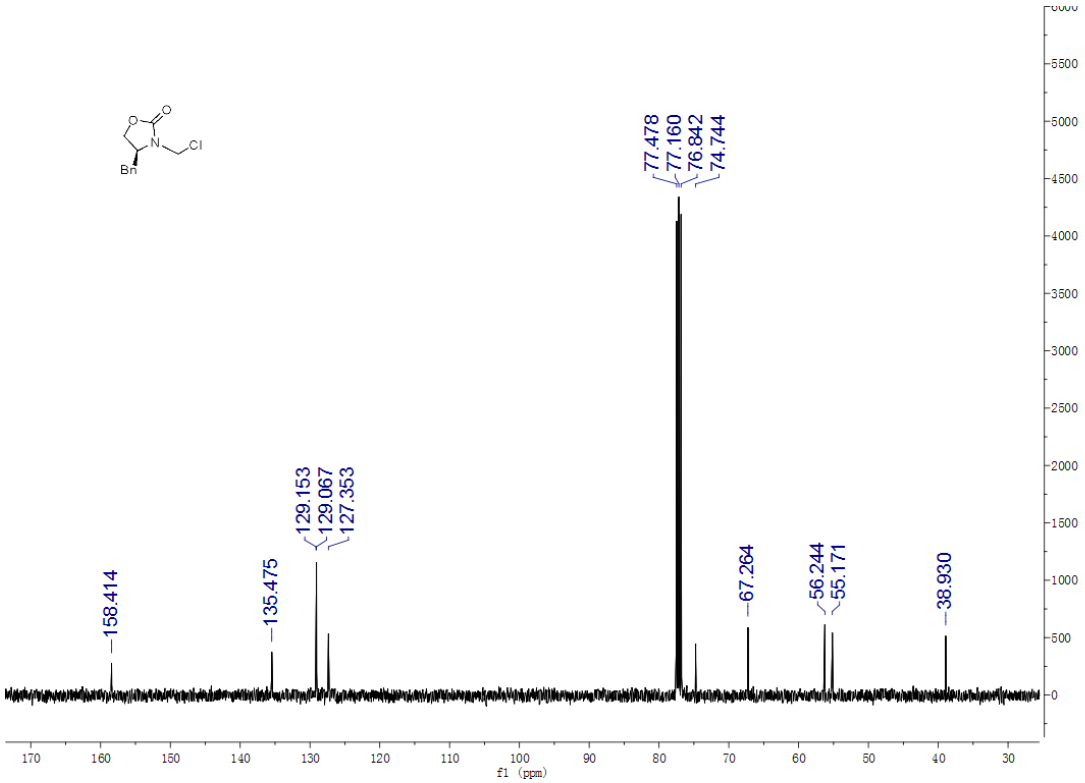

154.40
142.42
128.92
124.77
123.87
110.34
110.27
48.52
f1 (ppm)


7.260
4.898
4.208
4.195
4.181
3.454
3.439
3.423
1.977
1.962
1.950
1.947
1.940
1.935
1.920
2.00
4.07
4.02
4.11
154.287
67.024
62.072
45.088
22.066


7.40
7.32
7.27
7.11
6.36
4.97
4.02
4.06
2.00
4.00
f1 (ppm)


153.48
134.91
129.70
124.89
124.14
121.56
114.16
67.24
51.24
f1 (ppm)


7.365
7.348
7.329
7.303
7.285
7.280
7.267
7.203
7.185
4.873
4.845
4.712
4.685
4.232
4.212
4.176
4.083
4.069
4.064
3.299
3.288
3.264
3.255
2.739
2.716
2.705
2.683
3.14
1.97
1.00
1.02
2.02
1.03
1.04
1.02
f1 (ppm)


77.478
77.160
76.842
74.744
158.414
135.475
129.153
129.067
127.353
67.264
56.244
55.171
38.930
f1 (ppm)