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Supplementary Materials

Rapid Synthesis of Asymmetric Methyl-Alkyl Carbonates Catalyzed by α-$KMgPO_4$ in a Sealed-vessel Reactor Monowave 50

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The liquid products analyzed by gas chromatography (Figure S1), and analyzed by GC-MS spectrum (Figure S2 and Table S1). The main product (MOC and MLC) are final determined by the NMR characterization (Figure S3).

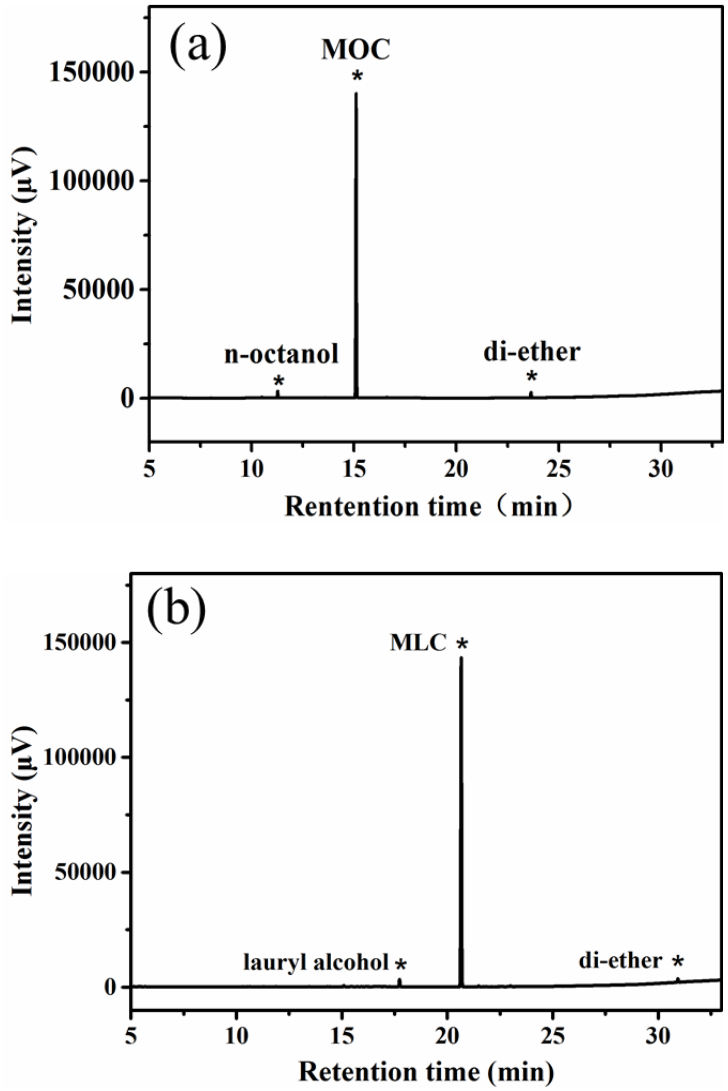


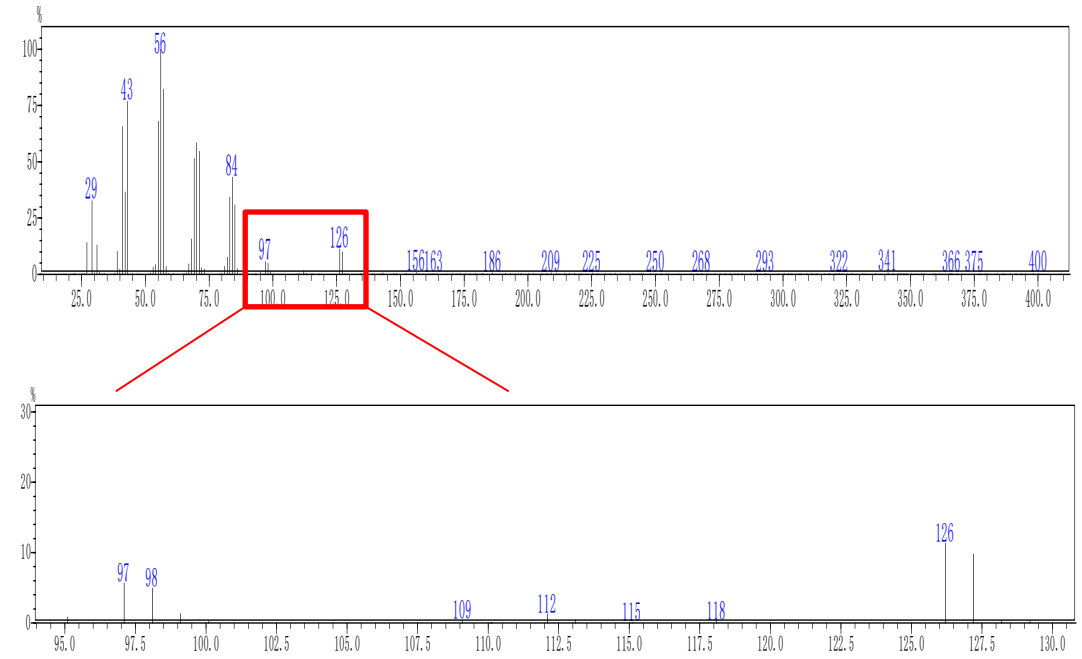
Figure S1. GC trace of the products: (**a**) MOC as the main product; (**b**) MLC as the main product.

The substances on GC all could correspond to the GC-MS, the corresponding fragment peak can be found on GC-MS spectrum. Figure S2a and Figure S2b show the GC-MS spectrum of the liquid products in MOC and MLC synthesis, respectively. The up is GC-MS spectrum in our instrument, and the bottom is the standard spectrum from Nist11.

Figure S2. GC-MS spectrum of the liquid products.

Figure S2a. GC-MS spectrum of the liquid product in MOC synthesis.

n-octanol: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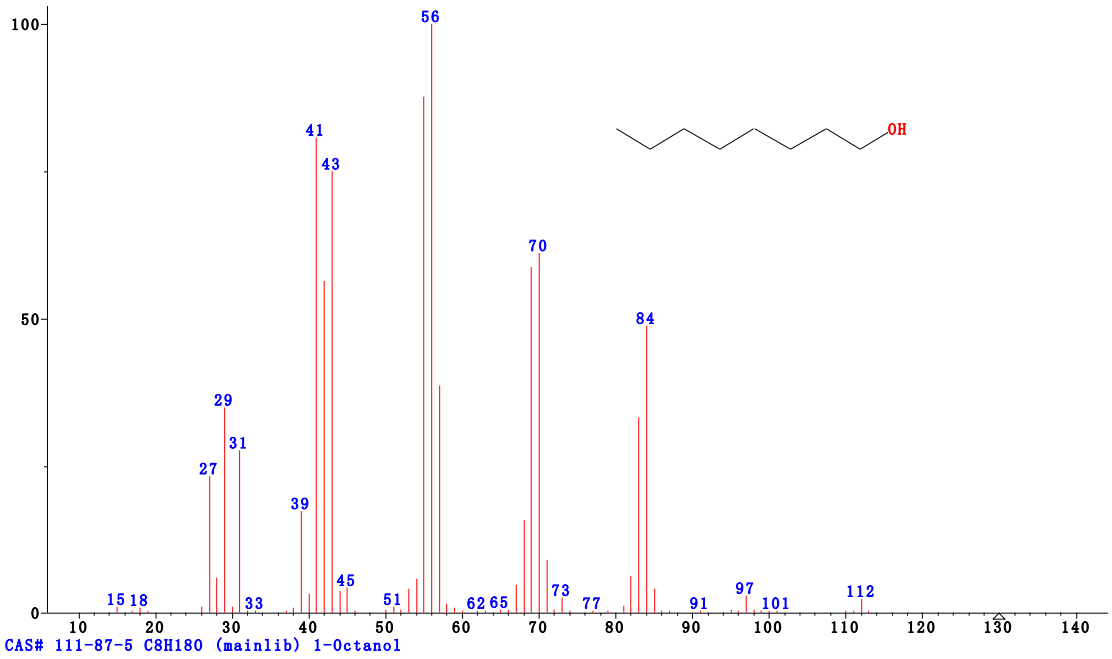
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41
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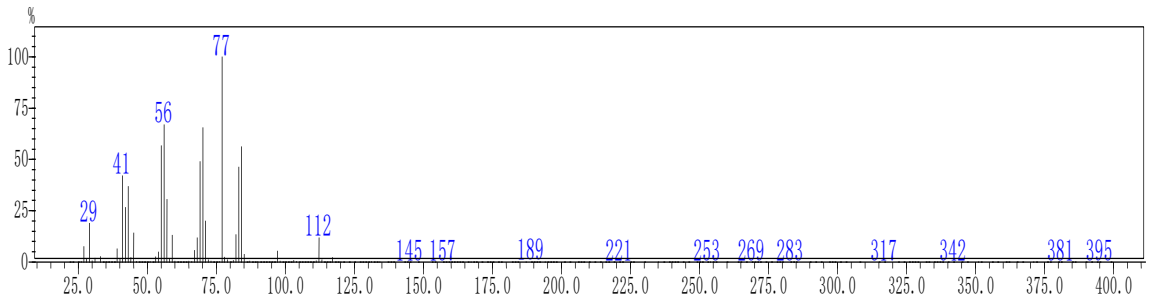
62 65
73
77
91
97
101
112
OH
10
20
30
40
50
60
70
80
90
100
110
120
130
140
CAS# 111-87-5 C8H18O (mainlib) 1-Octanol

MOC: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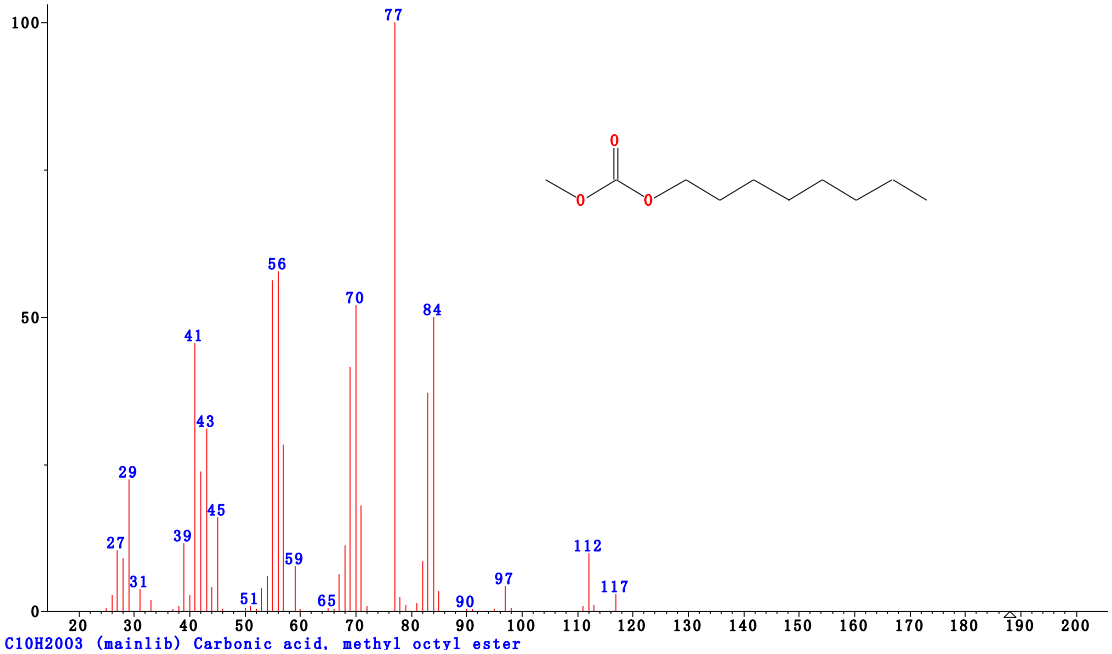
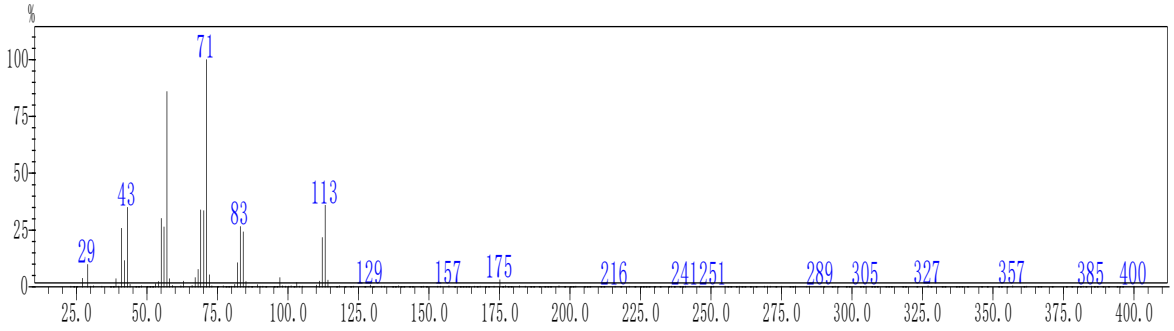


Dioctyl ether:



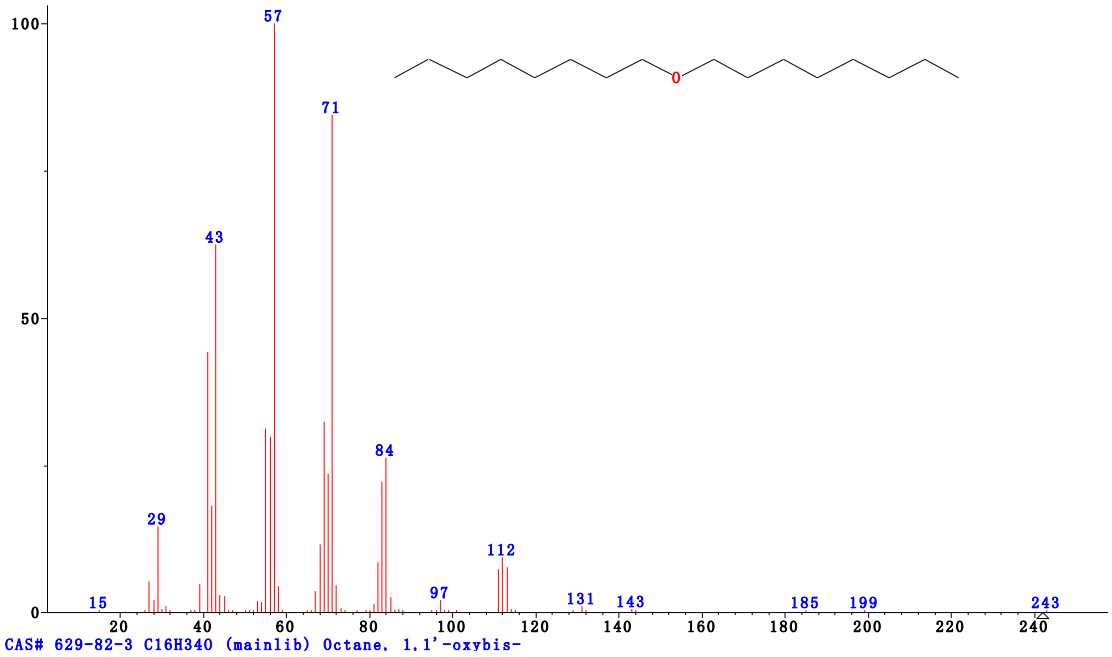
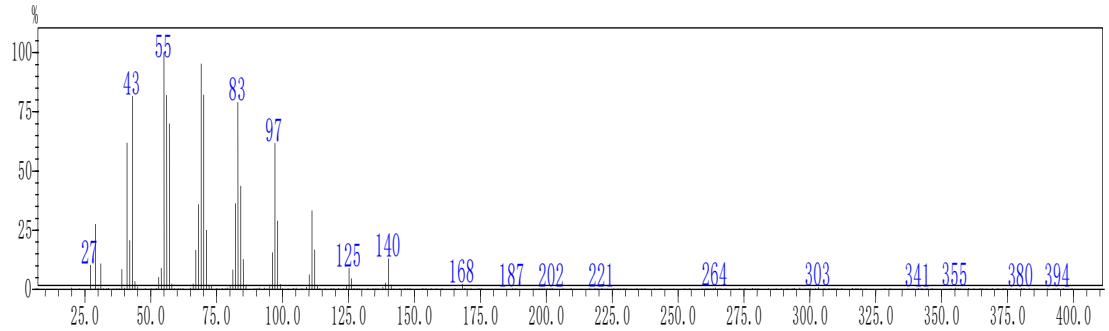
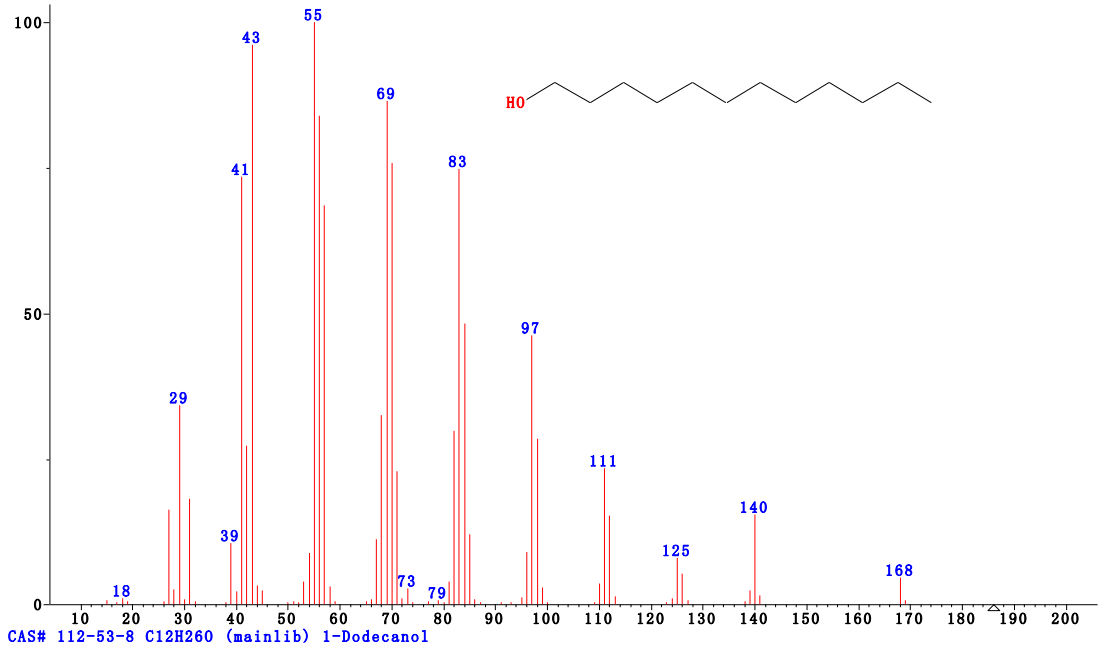


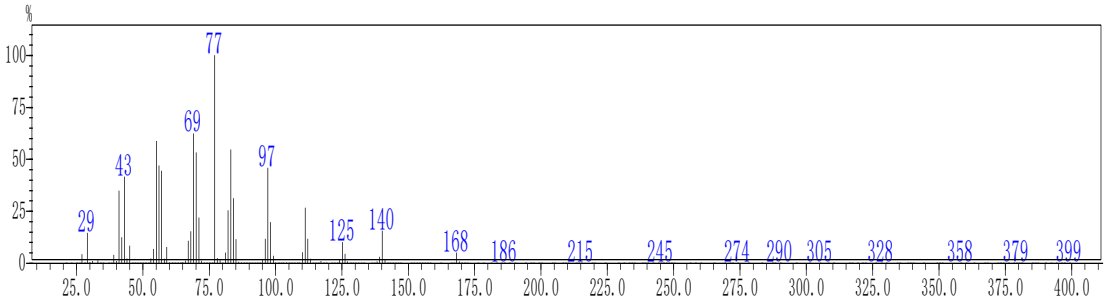
Figure S2b. GC-MS spectrum of the liquid product in MLC synthesis.

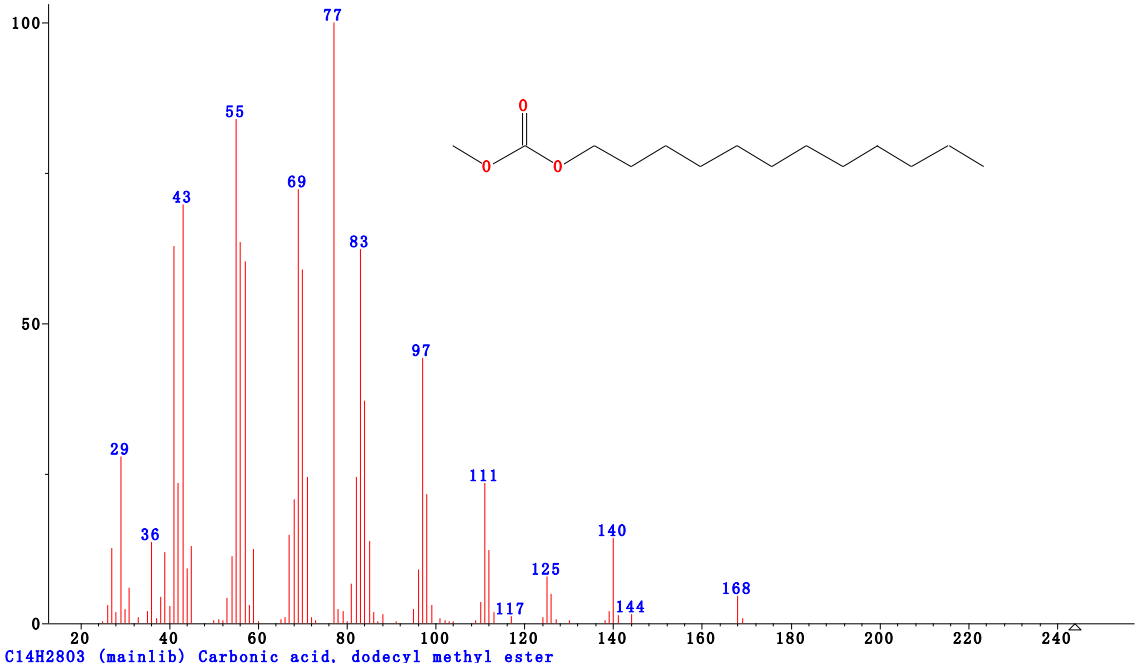
Lauryl alcohol:



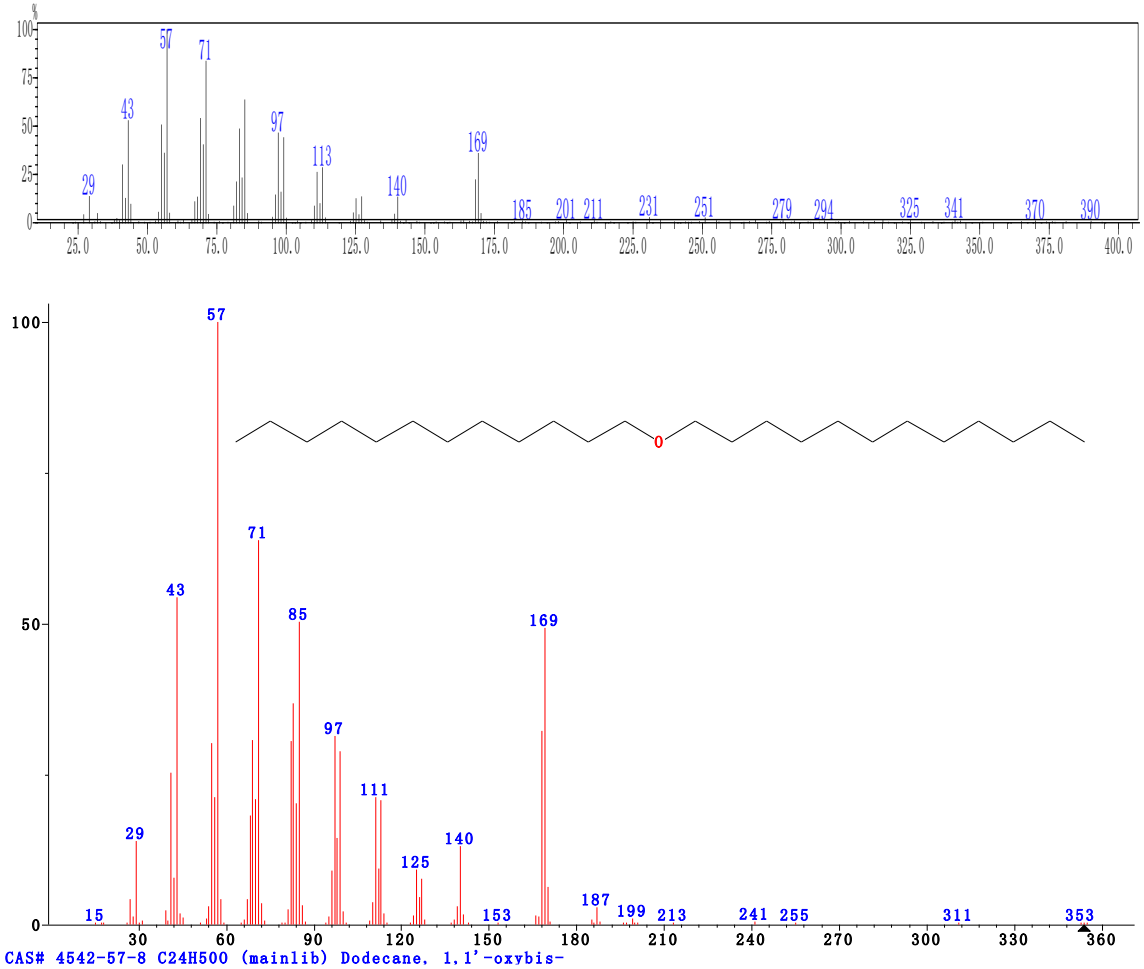


MLC:





Dilauryl ether:



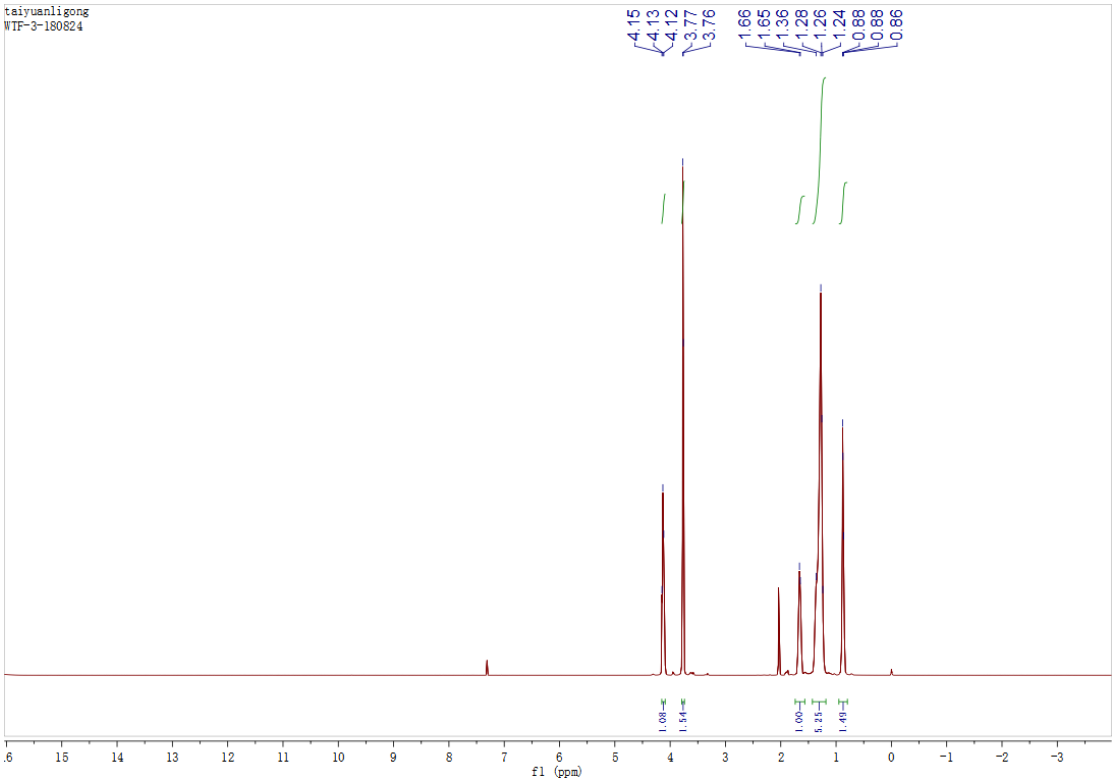
Particularly, the GC-MS data of the main product MOC and MLC are list in Table S1.

Table S1. Mass spectral data of MOC and MLC.

Substances	Characteristic mass spectral ions and abundances
MOC	77.05(100),56.05(66.98),70.1(65.54),55.05(56.66),84.1(56.17),69.1(48.95),83.1(46.34),41(41.96),43.05(36.78),57.05(30.56),42.05(26.66),71.1(20.03),29.05(18.84),45(14.21),82.1(13.38),112.15(11.86),68.05(12.78),97.1(5.28)
MLC	77.05(100),69.1(62.29),55.05(58.70),83.1(54.57),70.1(53.26),56.05(46.98),97.1(45.77),57.05(44.40),43.05(41.57),84.1(31.11),111.15(26.58),82.1(25.39),71.1(21.95),98.1(19.62),140.2(15.61),68.05(15.35),29.05(14.42),42.05(12.49),112.15(11.67),96.1(11.64),125.15(10.02)

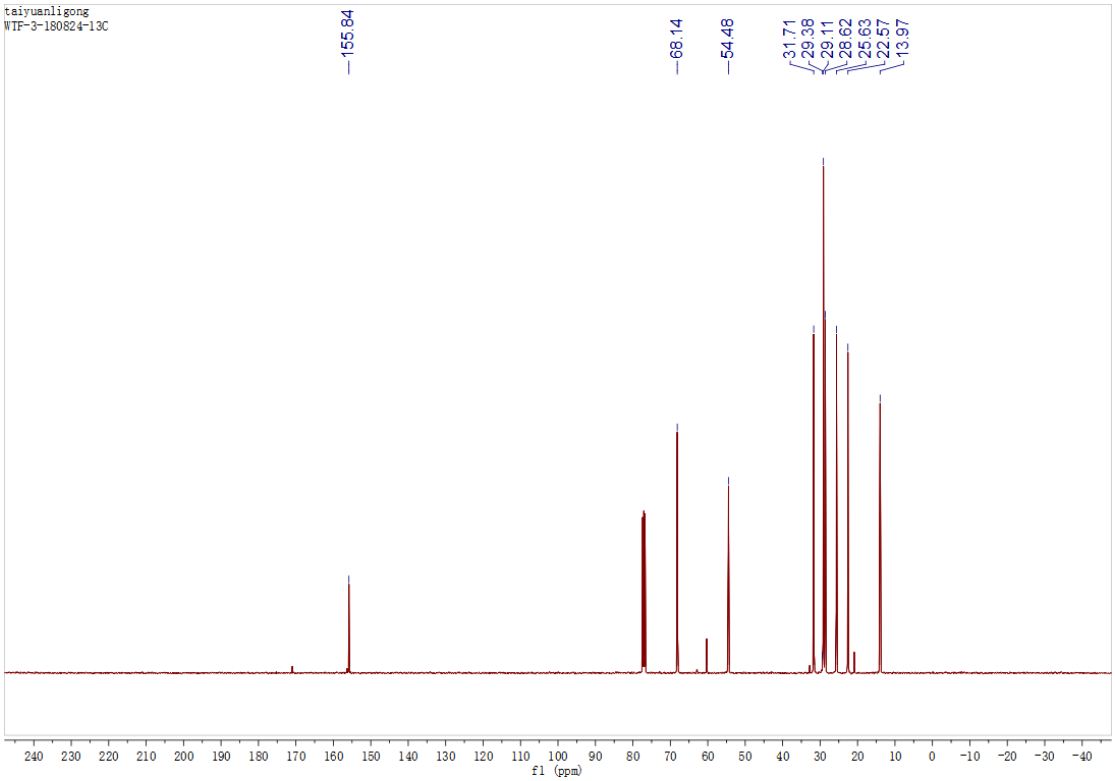
Figure S3. NMR Data of MOC and MLC.

Figure S3a. ^{1}H-NMR of MOC.



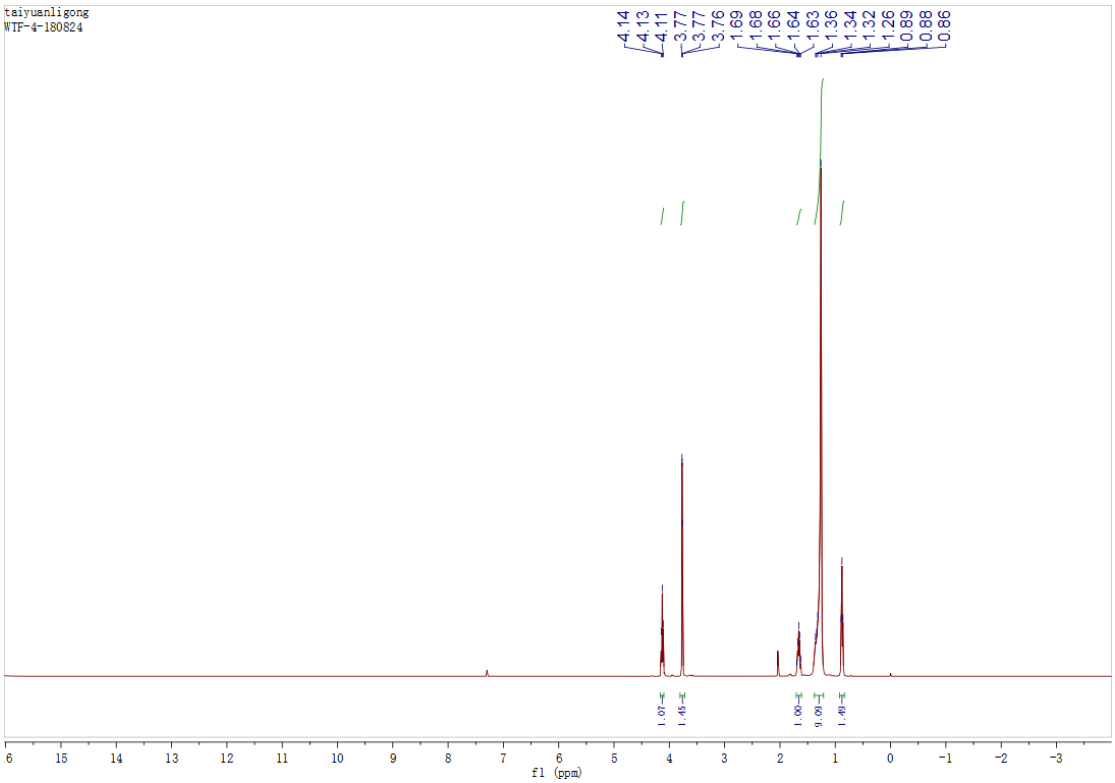
^{1}H NMR (400 MHz, $CDCl_3$) δ 4.13 (t, *J* = 6.6 Hz, 2H), 3.77 (d, *J* = 4.5 Hz, 3H), 1.65 (d, *J* = 6.2 Hz, 2H), 1.43 – 1.18 (m, 10H), 0.95 – 0.80 (m, 3H).

Figure S3b. ^{13}C-NMR of MOC.



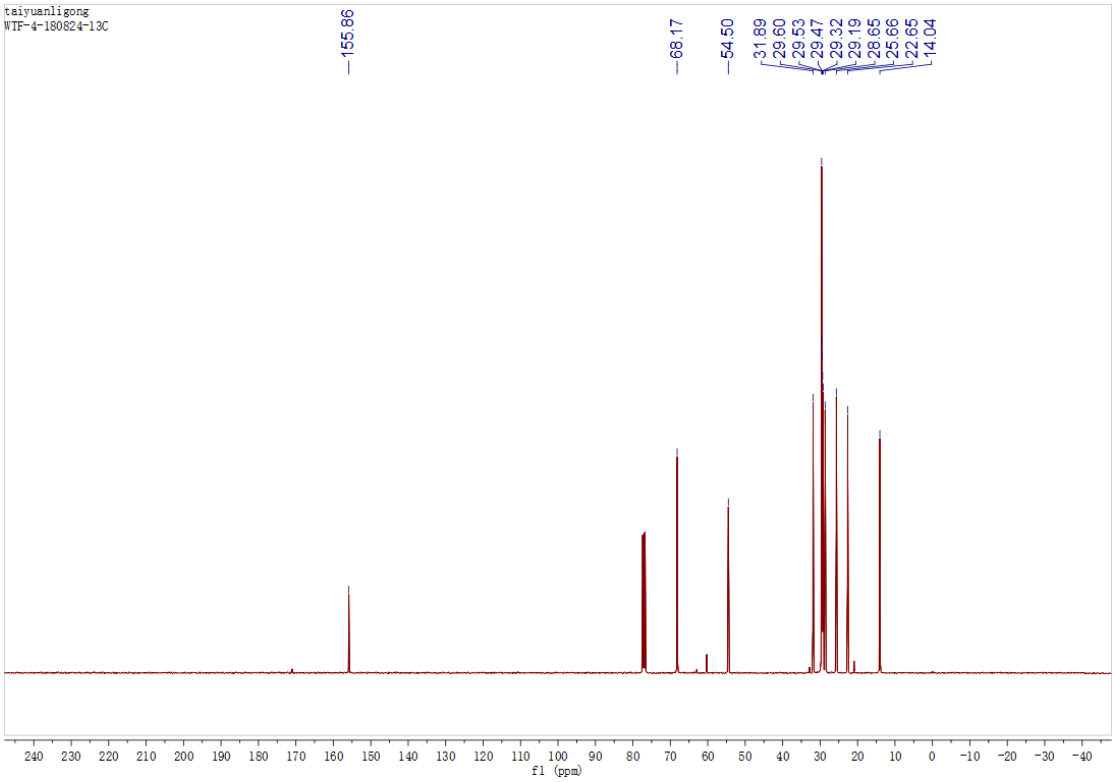
^{13}C NMR (101 MHz, $CDCl_3$) δ 155.84 (s), 68.14 (s), 54.48 (s), 31.71 (s), 29.38 (s), 29.11 (s), 28.62 (s), 25.63 (s), 22.57 (s), 13.97 (s).

Figure S3c. ^{1}H-NMR of MLC.



^{1}H NMR (400 MHz, $CDCl_3$) δ 4.13 (t, *J* = 6.7 Hz, 2H), 3.81 – 3.72 (m, 3H), 1.70 – 1.61 (m, 2H), 1.38 – 1.21 (m, 18H), 0.88 (t, *J* = 6.3 Hz, 3H).

Figure S3d. ^{13}C-NMR of MLC.



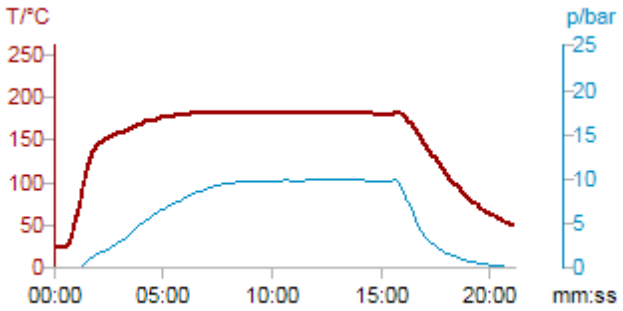
^{13}C NMR (101 MHz, $CDCl_3$) δ 155.86 (s), 68.17 (s), 54.50 (s), 31.89 (s), 29.95 – 28.87 (m), 28.65 (s), 25.66 (s), 22.65 (s), 14.04 (s).

Figure S4. The parameters recorded on Monowave 50 reactor.

The reaction temperature can be controlled accurately by Monowave 50 reactor, the changing curve of temperature and pressure were recorded. Figure S4 shows the complete process under optimal reaction conditions.

High efficent thermal experiment with simulating microwave irradiation

- Anton Paar Monowave 50
- Serial Number 81991167/0
- Instrument Software Version 1.10.3211.2



- Date and Time 11/9/2018 10:06:27
- User Administrator

- Method Name Quick

- Steps

No	Step Type	Temp (°C)	Time (hh:mm)
1	AFAP	180	-
2	Hold Time	180	0:10

- Stirrer Speed 1000
- Cooling Temperature 50

- Result

Description	Value
Result	OK
Condition	-
Emergency	-
Total Runtime	00:21:12 (hh:mm:ss)
First T-Setpoint Reached	00:05:32 (hh:mm:ss)
Max. Temperature	182

The specific details of the steel plate and steel ball used:

An AISI 52100 steel ball (diameter 10 mm, HRC 59-64, Ra 0.014 μm) over an AISI 52100 steel disk (24 mm diameter, 7.85 mm height, HRC 59-61, Ra 0.124 μm) were used in test. Before surface analysis, the test specimens were washed by immersion in petroleum ether so as to eliminate all of the residual oil and the wear debris.