

Supplementary Materials: Design and Development of Novel Continuous Flow Stirred Multiphase Reactor: Liquid-Liquid-Liquid Phase Transfer Catalysed Synthesis of Guaiacol Glycidyl Ether

Nikhil H. Margi and Ganapati D. Yadav

1. Reactor photo and diagrams

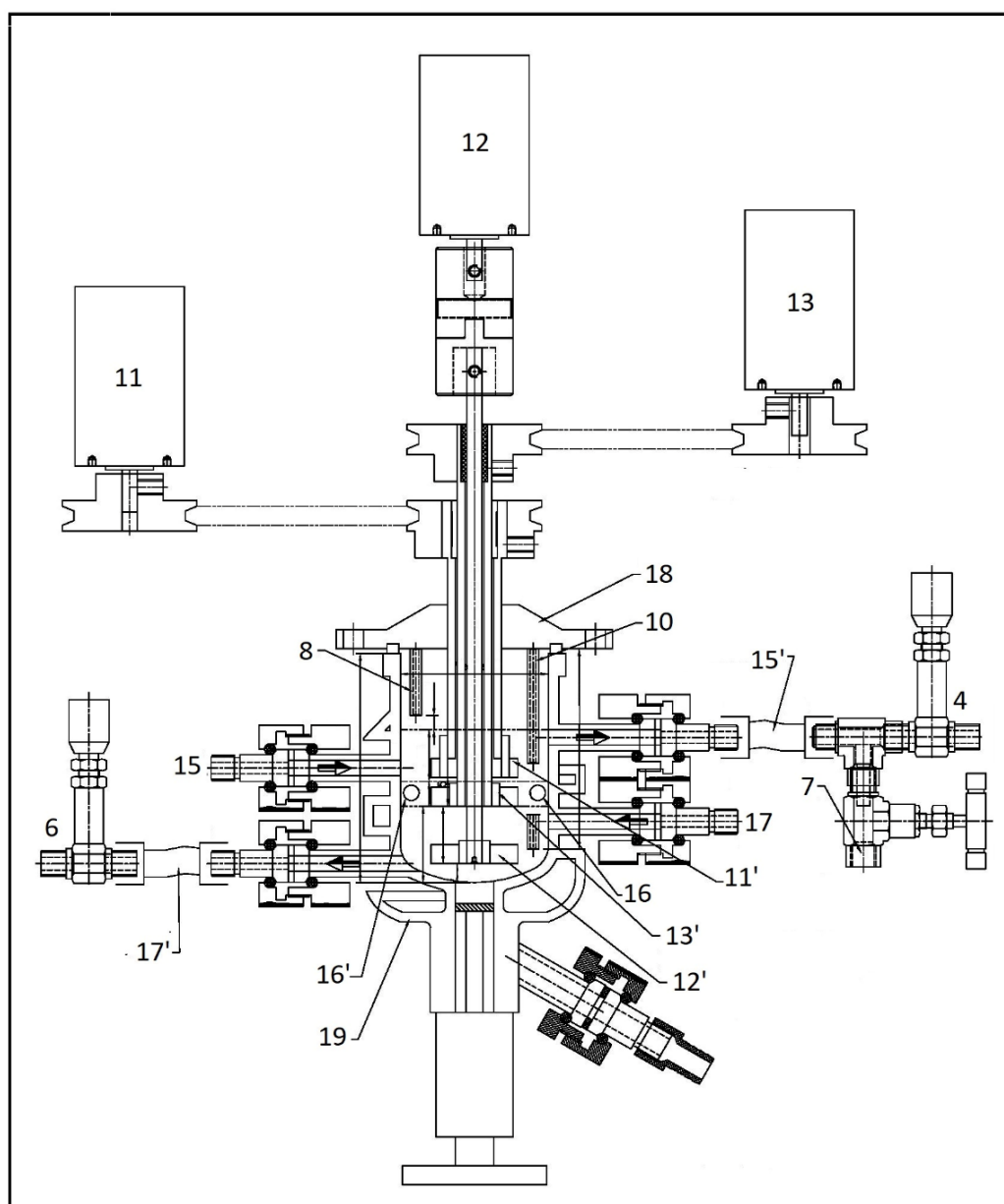


Figure 1. Schematic diagram of the front section the reactor vessel.

4- organic phase metering valve, 6- aqueous phase metering valve, 7- sampling valve for organic phase, 8- overflow tube, 10 adjustable sampling tube with feed system, 11- motor for organic phase, 12- motor for aqueous

phase, 13- motor for middle phase, 11'- stirrer for organic phase, 12'- stirrer for aqueous phase, 13'-stirrer for middle phase, 15- inlet for organic phase, 16- inlet for middle phase, 17- inlet for aqueous phase, 15'- outlet for organic phase, 16'- outlet for middle phase, 17'- outlet for aqueous phase, 18- jacketed heating system, 19- drain valve.

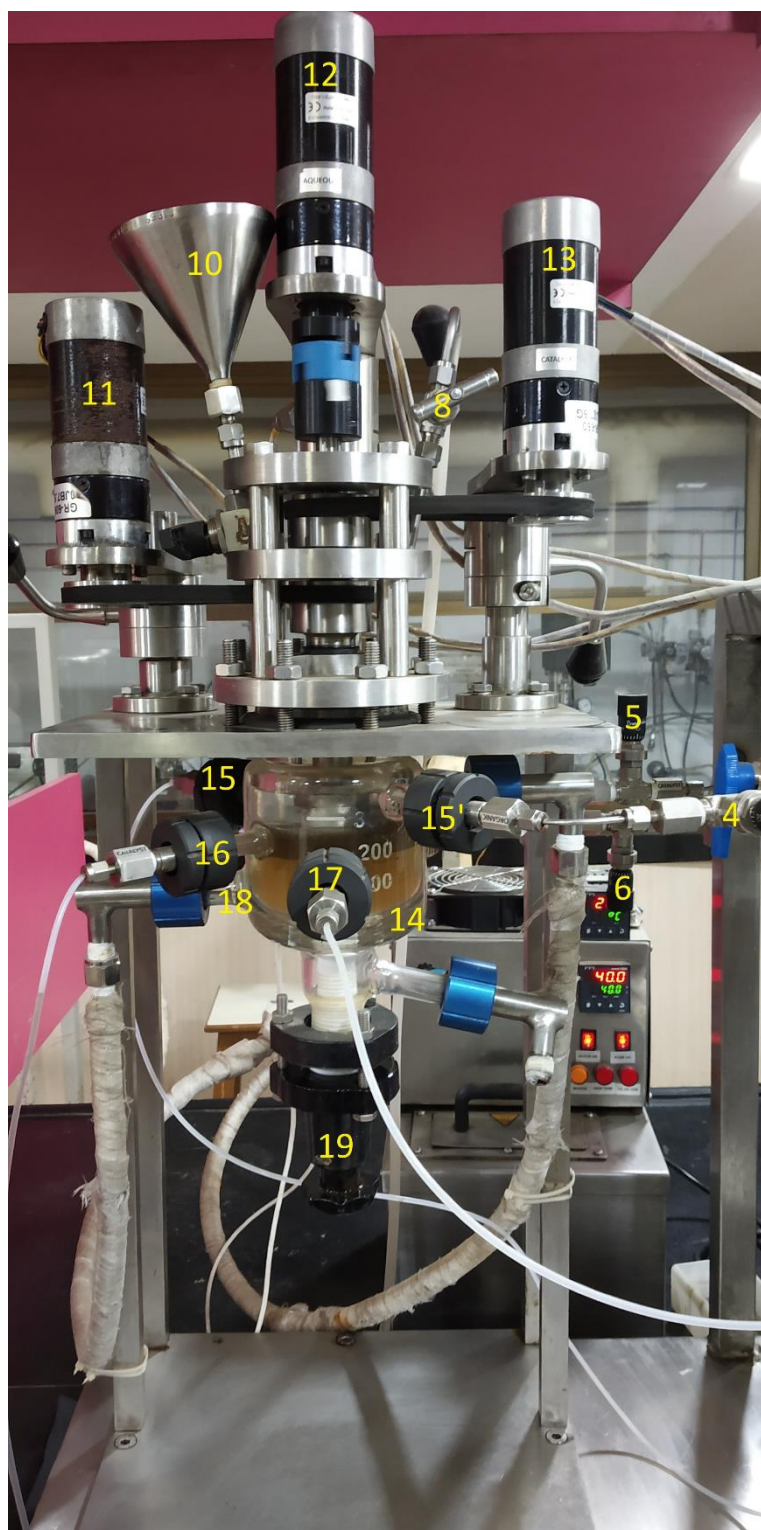


Figure 2. Front view of the actual reactor vessel.

4- organic phase metering valve, 5- middle phase metering valve, 6- aqueous phase metering valve, 8- overflow tube, 10 adjustable sampling tube with feed system, 11- motor for organic phase, 12- motor for aqueous phase, 13- motor for middle phase, 14- reactor glass vessel, 15- inlet for organic phase, 16- inlet for middle phase, 17- inlet for aqueous phase, 15'- outlet for organic phase, 18- jacketed heating system, 19- drain valve.

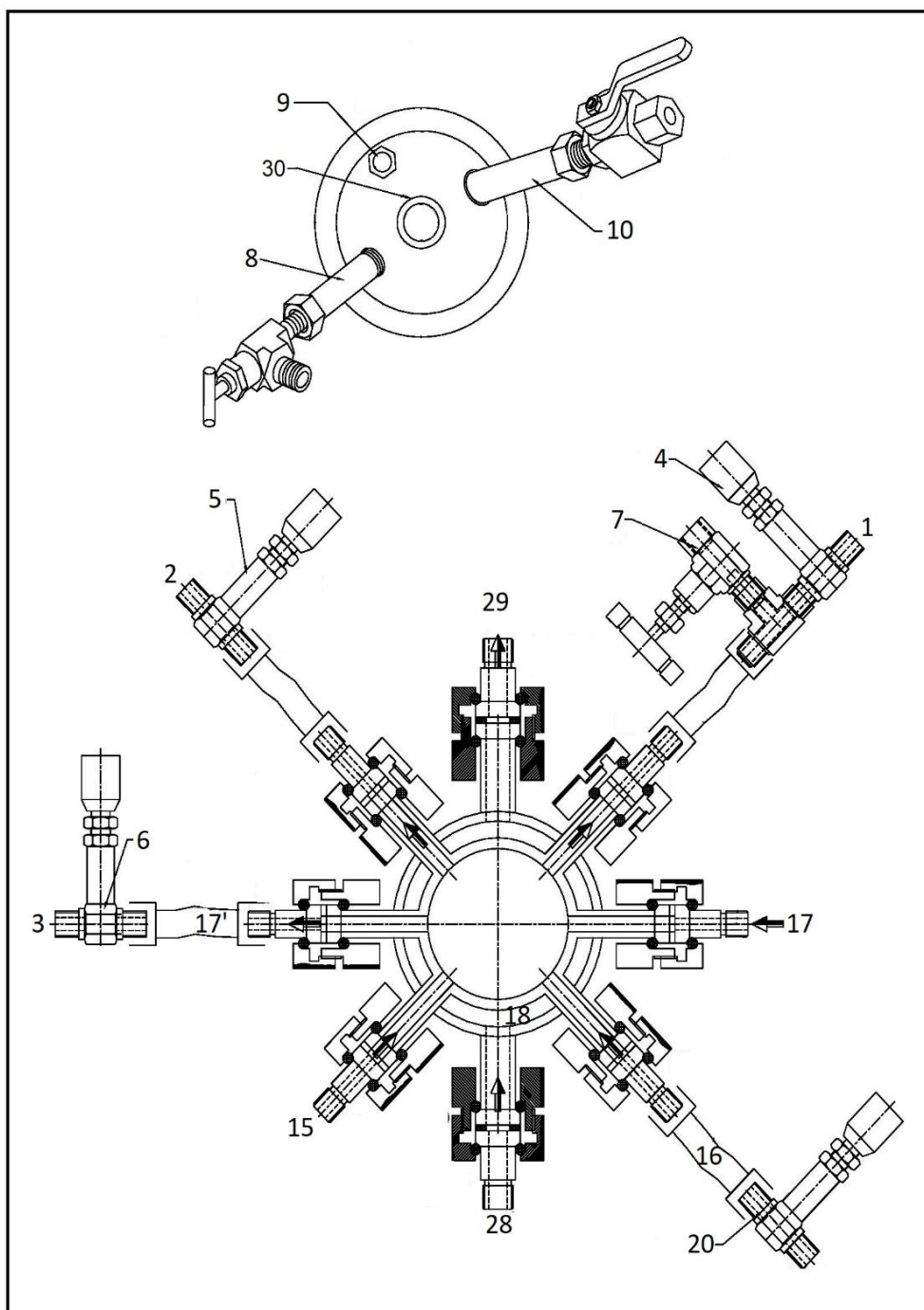


Figure 3. Schematic diagram of a top view of the reactor vessel.

1-outlet for organic phase metering valve, 2- outlet for middle phase metering valve, 3- outlet for aqueous phase metering valve, 4- organic phase metering valve, 5- middle phase metering valve, 6- aqueous phase metering

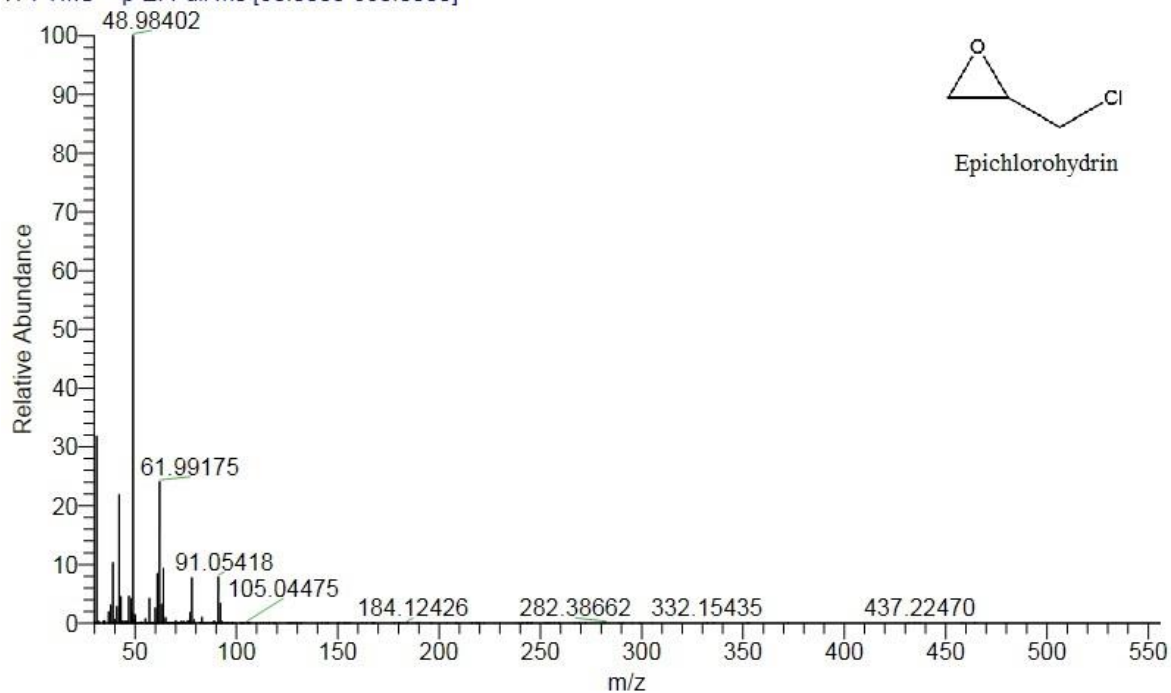
valve, 7- sampling valve for organic phase, 8- overflow tube, 9- thermowell with thermocouple, 10 adjustable sampling tube with feed system, 15- inlet for organic phase, 16- inlet for middle phase, 17- inlet for aqueous phase, 15'- outlet for organic phase, 16'- outlet for middle phase, 17'- outlet for aqueous phase, 18- jacketed heating system, 20- metering valve at middle phase inlet, 28- jacket heating media in, 29- jacket out heating media out, 30- stirrer assembly hole.

2. GCMS analysis

The Thermo Scientific Q Exactive Orbitrap GC-MS (HRMS) was used for the confirmation of the product. Following were the MS spectrums for all the GC peaks including A) Epichlorohydrin, B) Guaiacol glycidyl ether, C) reaction by-product and D) n-decane.

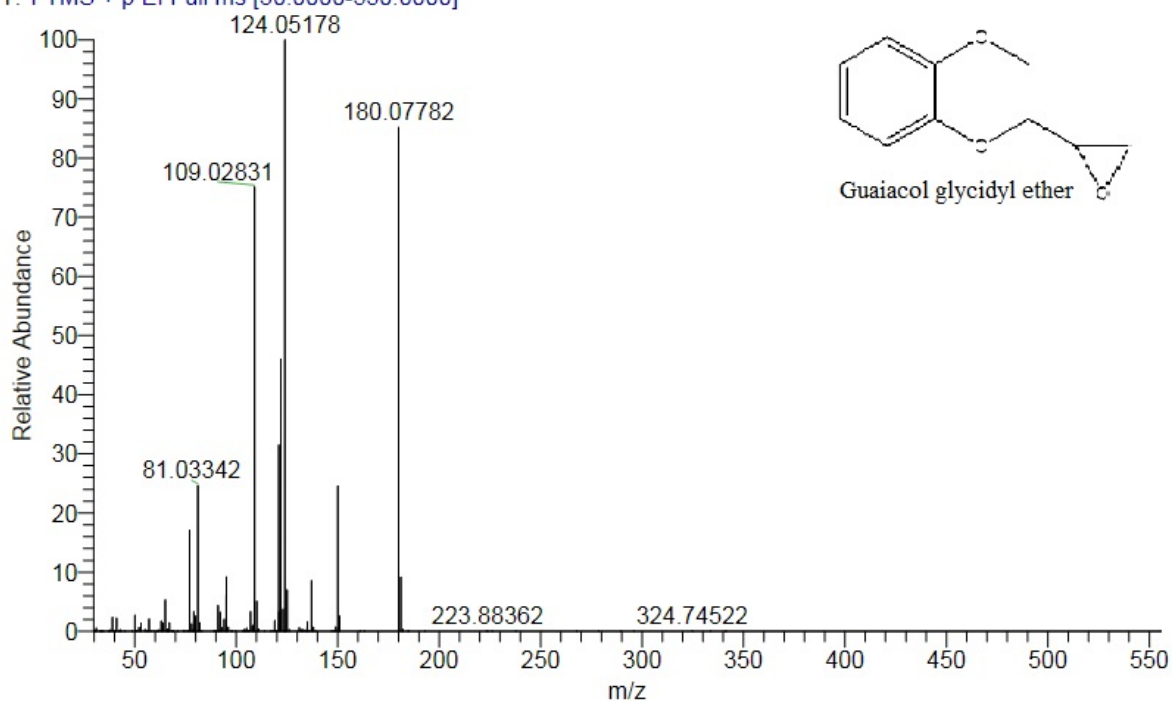
A) Epichlorohydrin

PTC3Phase_G+E_Cont3 #437 RT: 1.96 AV: 1 SB: 40 13.66-13.75 , 13.87-13.95 NL: 1.83E8
T: FTMS + p EI Full ms [30.0000-550.0000]



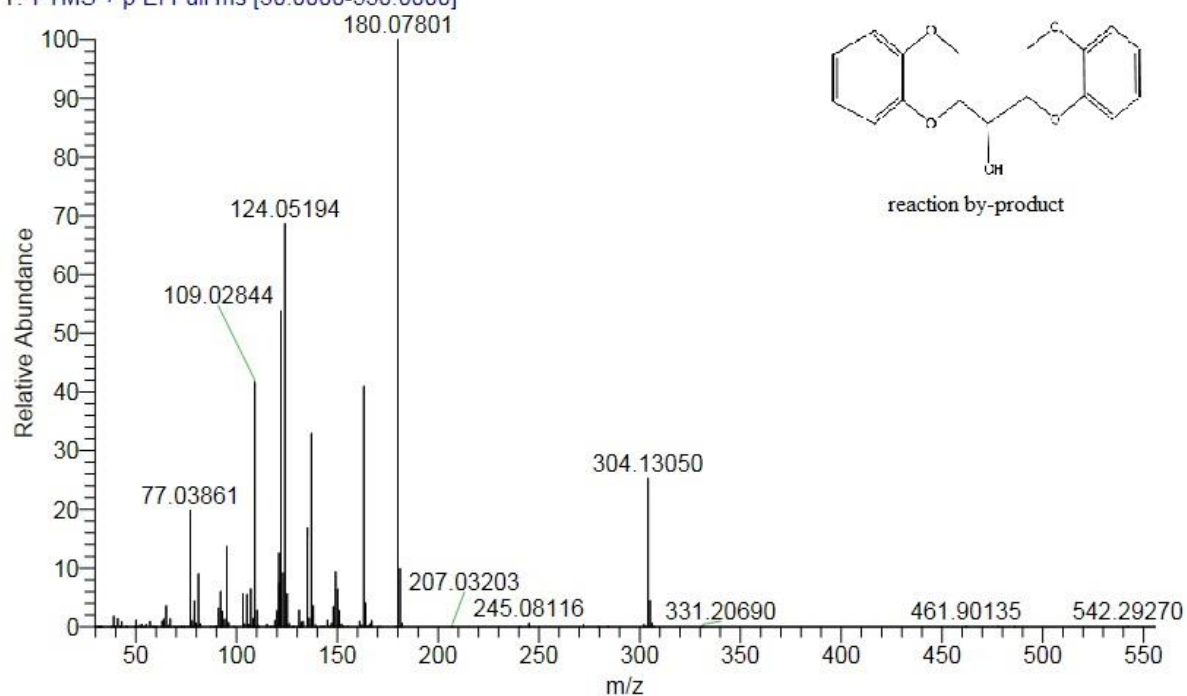
B) Guaiacol glycidyl ether

PTC3Phase_G+E_Cont3 #1876 RT: 8.43 AV: 1 SB: 40 13.66-13.75, 13.87-13.95 NL: 3.14E9
T: FTMS + p EI Full ms [30.0000-550.0000]



C) Reaction by-product

PTC3Phase_G+E_Cont3 #2926 RT: 13.14 AV: 1 SB: 40 13.66-13.75, 13.87-13.95 NL: 1.41E9
T: FTMS + p EI Full ms [30.0000-550.0000]



D) n-decane.

PTC3Phase_G+E_Cont3 #952 RT: 4.28 AV: 1 SB: 40 13.66-13.75, 13.87-13.95 NL: 1.91E9
 T: FTMS + p EI Full ms [30.0000-550.0000]

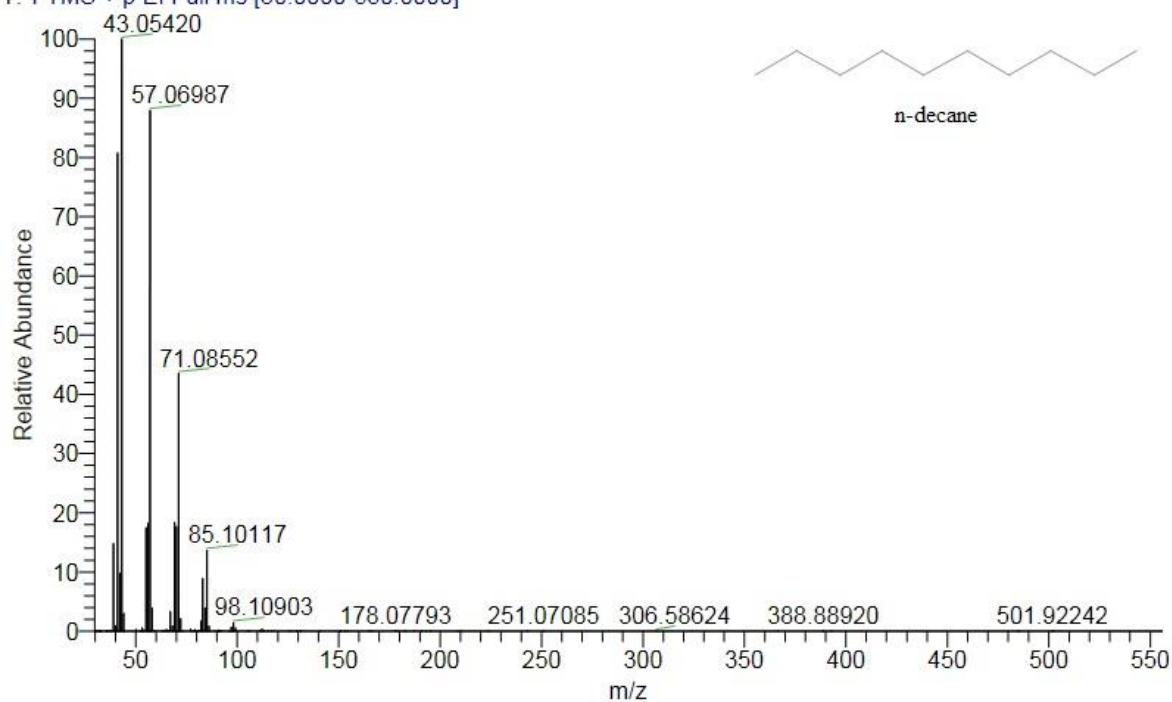


Figure 4. GCMS spectra of A) Epichlorohydrin, B) Guaiacol glycidyl ether, C) reaction by-product and D) n-decane

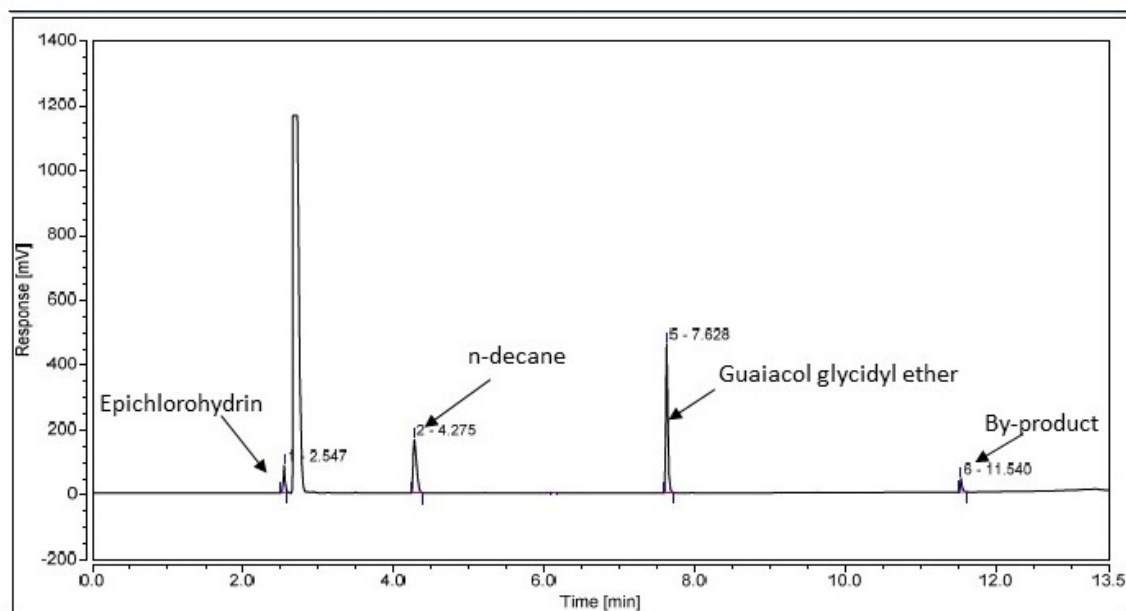


Figure 5. Typical GC chromatogram of reaction mass.