Synthesis and characterization of a new reusable calix[4]arene-bonded silica gel sorbent for antidiabetic drugs

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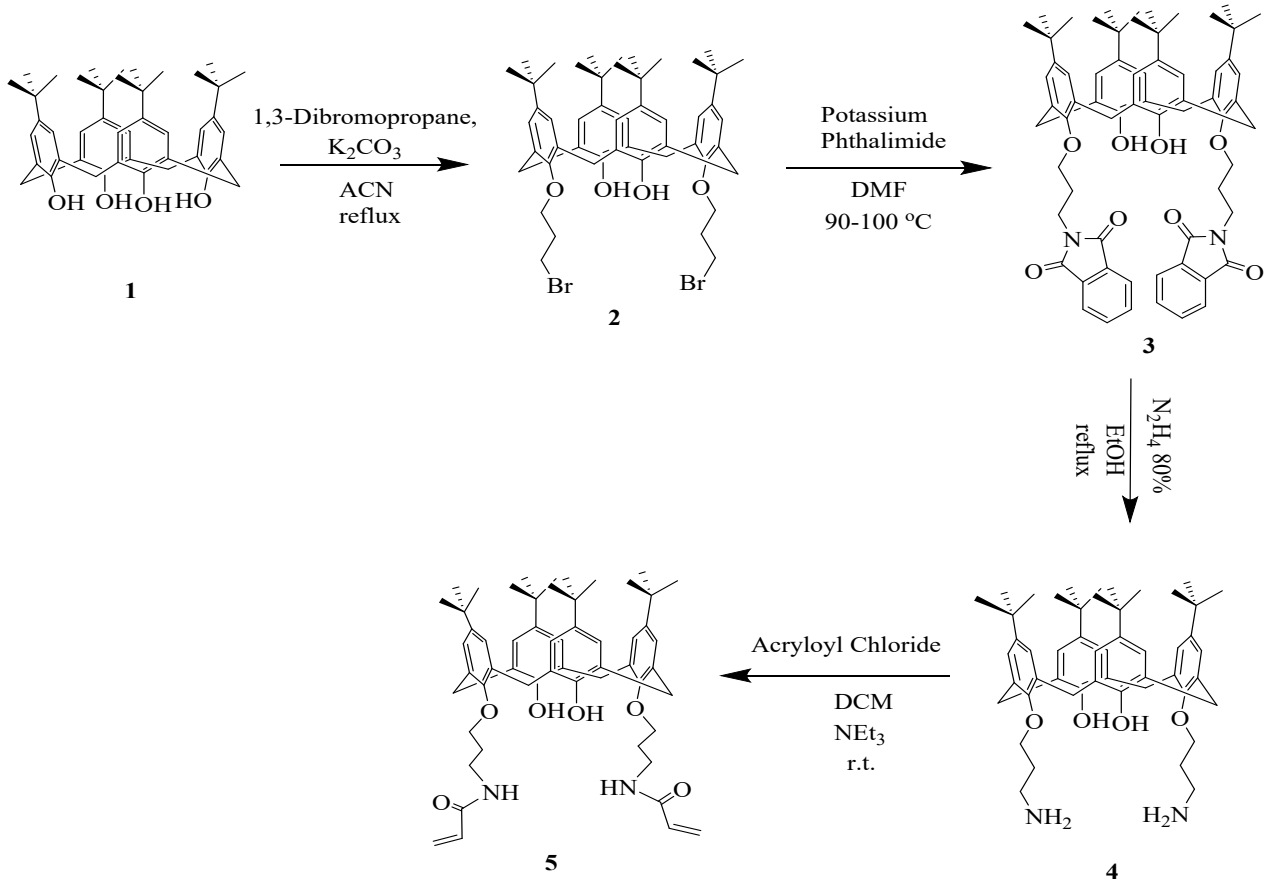
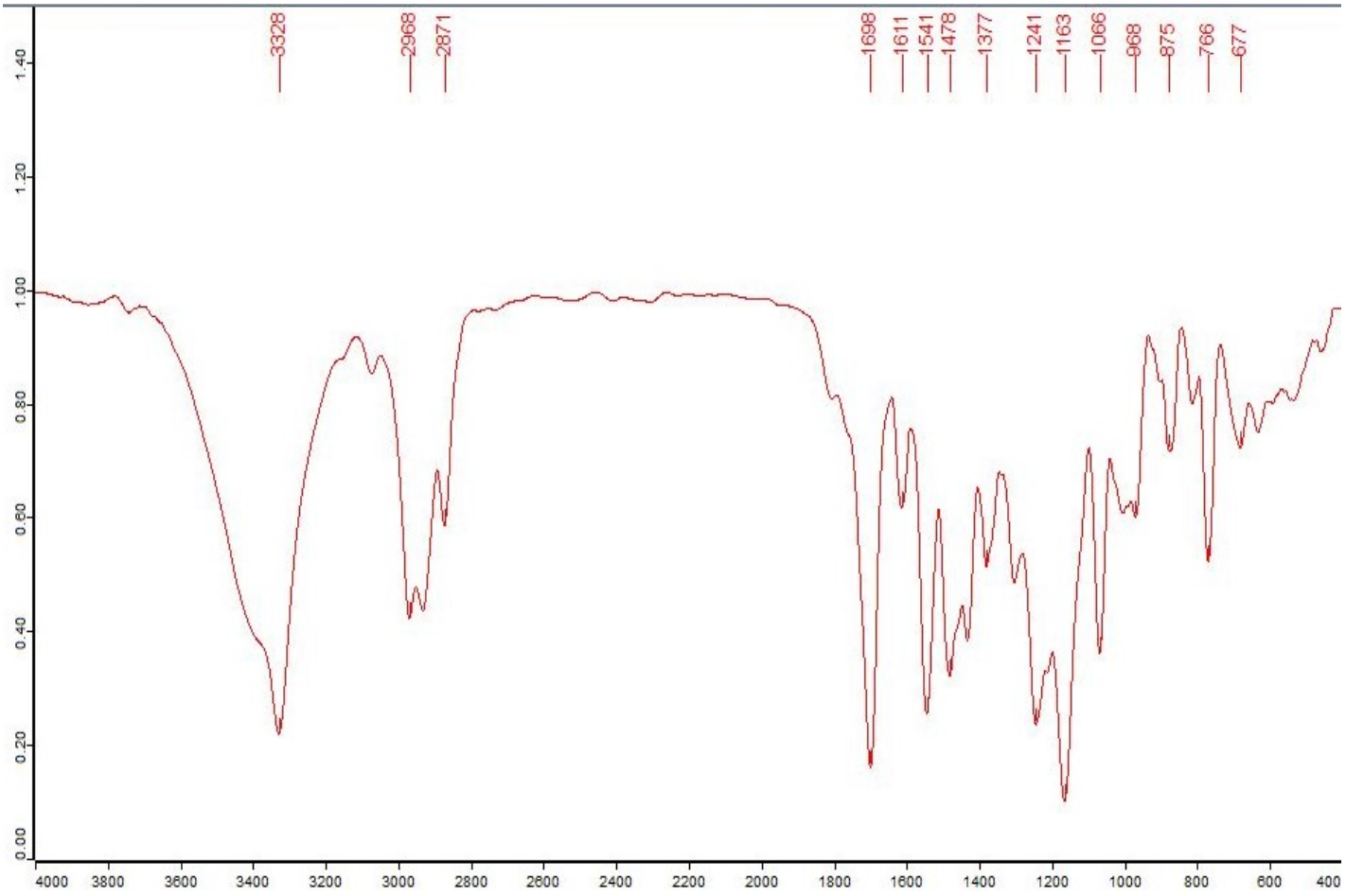
SUPPLEMENTARY INFORMATION

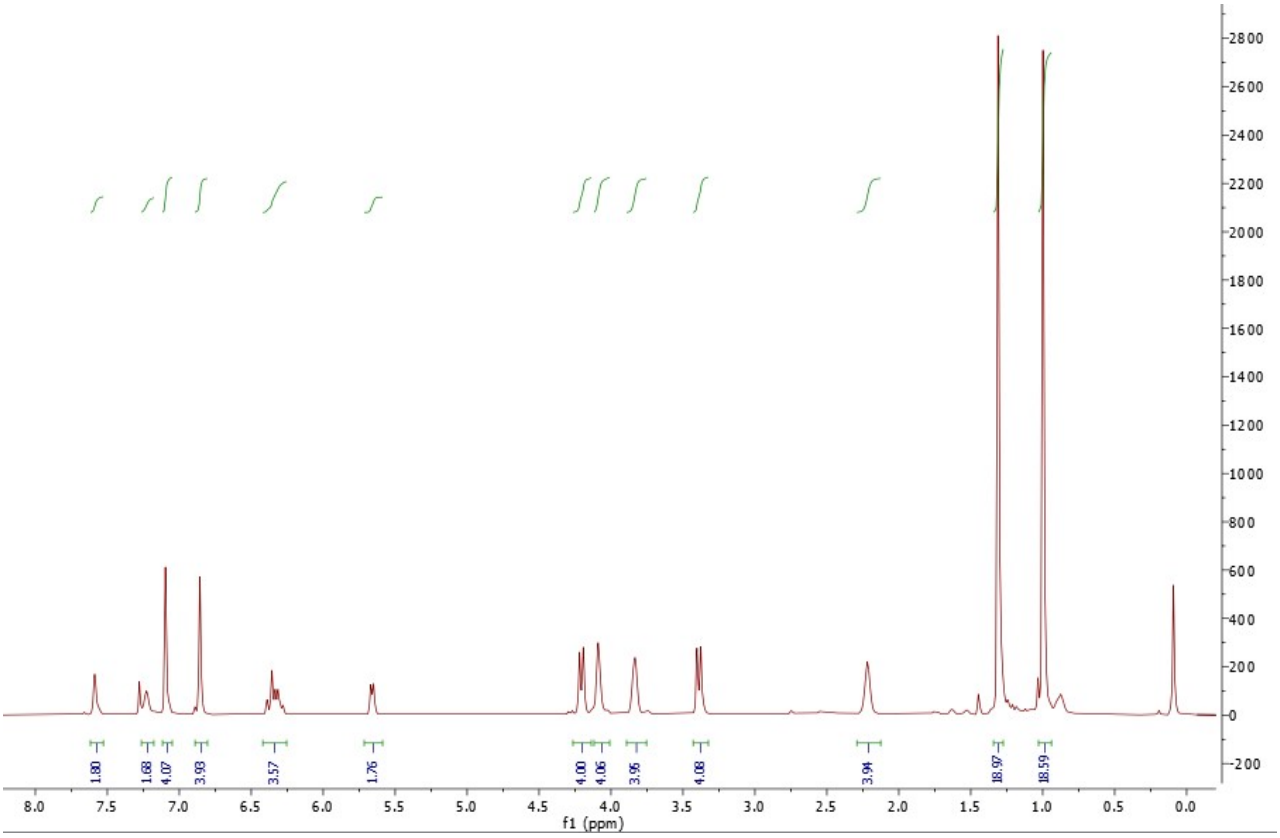
5,11,17,23-Tetra-tert-butyl-25,27-bis(N-acrylo-3-aminopropoxy)-26,28-dihydroxy-calix[4]arene (5)

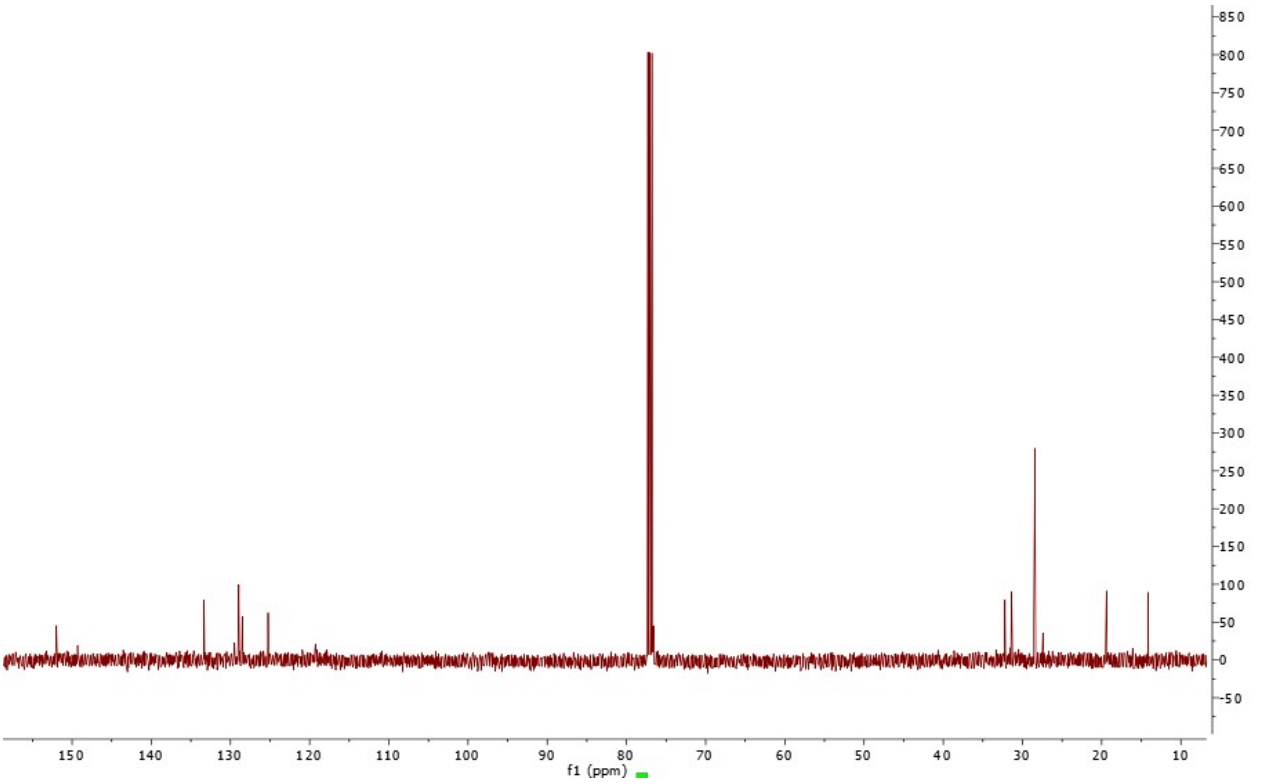
Acryloyl chloride (0.16 mL, 2.0 mmol) and triethylamine (0.35 ml, 2.0 mmol) were added to a solution of **4** (0.8 g, 1.0 mmol) in anhydrous THF (10 ml). The mixture was stirred under nitrogen atmosphere at -10 °C for 2 h and mixing was continued for another 24 h at room temperature. The mixture was concentrated under reduced pressure at 70 °C, the precipitate was dissolved in CH_2Cl_2 and was washed twice with water. The organic layer was dried with sodium sulfate and then was concentrated under reduced pressure to give a solid residue. The solid was recrystallized from methanol to give 0.57 g of **5,** as a light-yellow powder: yield 65%; mp: 251-253 °C; FT-IR (KBr, ν, cm^{-1}): 3328, 2968, 2871, 1698, 1611, 1541, 1478, 1377, 1241, 1163, 1066, 968, 875, 766, 677; ^1H NMR: (500 MHz, $CDCL_3$, TMS), δ (ppm), 1.12 (18H, s, $C(CH_3)_3$), 1.31 (18H, s, $C(CH_3)_3$), 2.24 (4H, m, $OCCH_2$), 3.39 (4H, d, (CH_2) eq-bridge, J = 12.1), 4.25 (4H, d, (CH_2) ax-bridge, J = 12.1), 3.81 (4H, unresolved t , NCH_2), 4.15 (4H, unresolved t , OCH_2), 5.68 (2H, d, CH-olefin, J = 9.3), 6.41 (4H, m, CH_2-olefin), 6.81 (4H, s, CH aromatic), 7.23 (4H, s, CH aromatic), 7.29 (2H, s, NH-amid), 7.61 (2H, s, OH-phenol); ^{13}C NMR: (125 MHz, $CDCl_3$), δ (ppm), 152.08, 149.27, 133.16, 129.65, 129.10, 128.31, 125.18, 76.44, 32.23, 31.22, 28.40, 27.39, 19.40.


1,3-Dibromopropane,
K_2CO_3
ACN
reflux
Potassium
Phthalimide
DMF
90-100 ºC
N_2H_4 80%
EtOH
reflux
Acryloyl Chloride
DCM
NEt_3
r.t.

1 **2** **3** **4** **5**


3328
2968
2871
1698
1611
1541
1478
1377
1241
1163
1066
968
875
766
677
1.40
1.20
1.00
0.80
0.60
0.40
0.20
0.00
4000
3800
3600
3400
3200
3000
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
400


1.80
1.68
4.07
3.93
3.57
1.76
4.00
4.06
3.96
4.08
3.94
18.97
18.59
f1 (ppm)


f1 (ppm)