Catalytic metallodendrimer grafted on mesoporous polymethacrylate beads for regioselective synthesis of β-amino alcohols under solvent-free conditions

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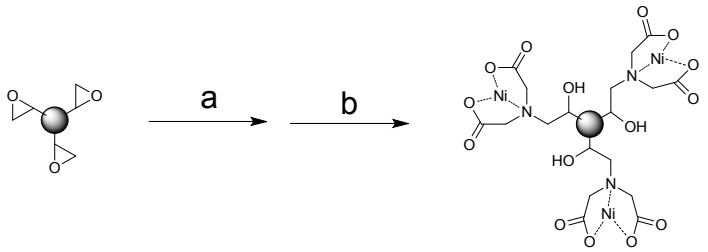
Supplementary Information

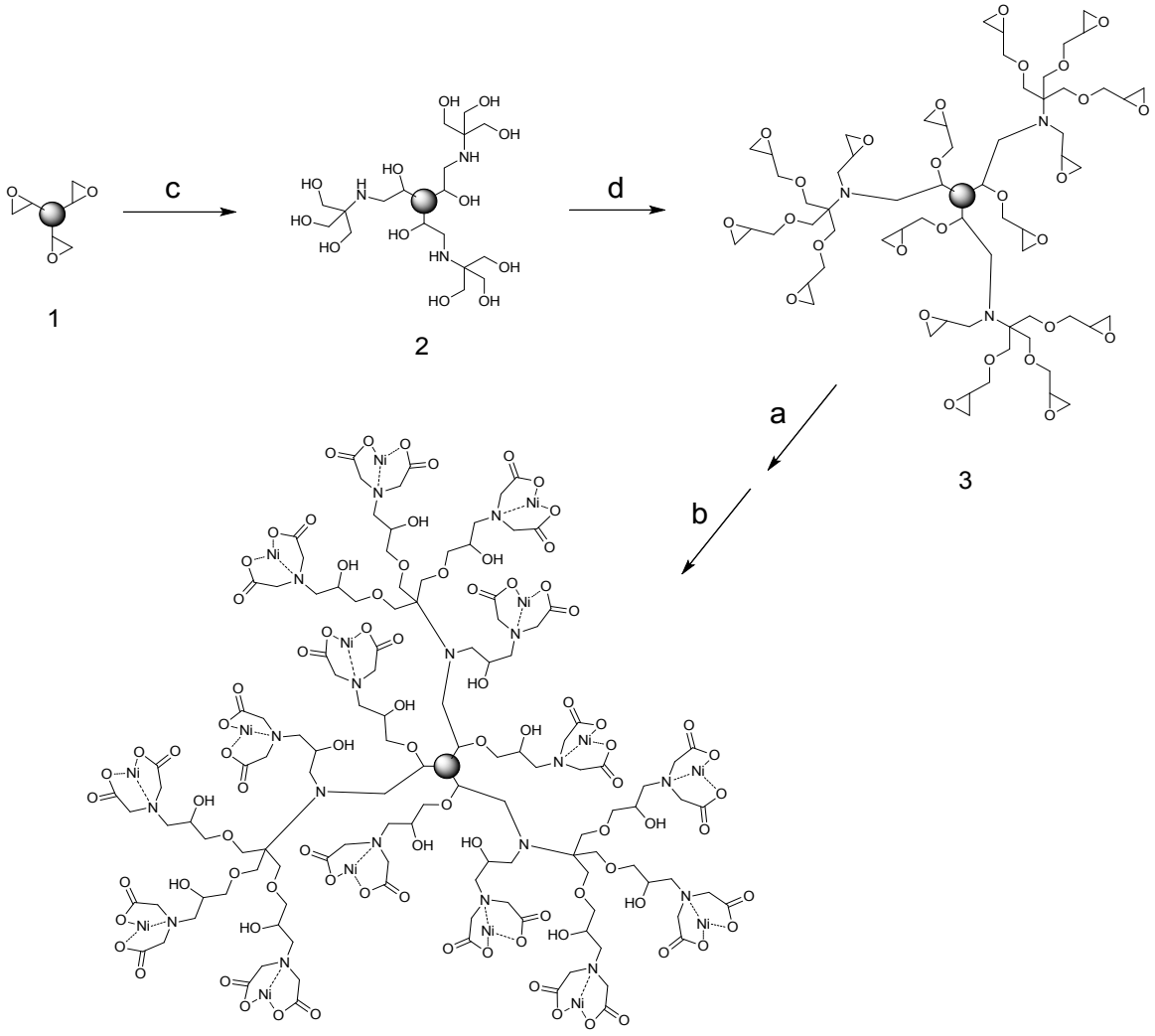
A) Scheme 2: Scheme for preparation G0, G1 and G2 series of metallodendritic side groups grafted polymethacrylate sepabeads catalysts

Series G0



1 G0

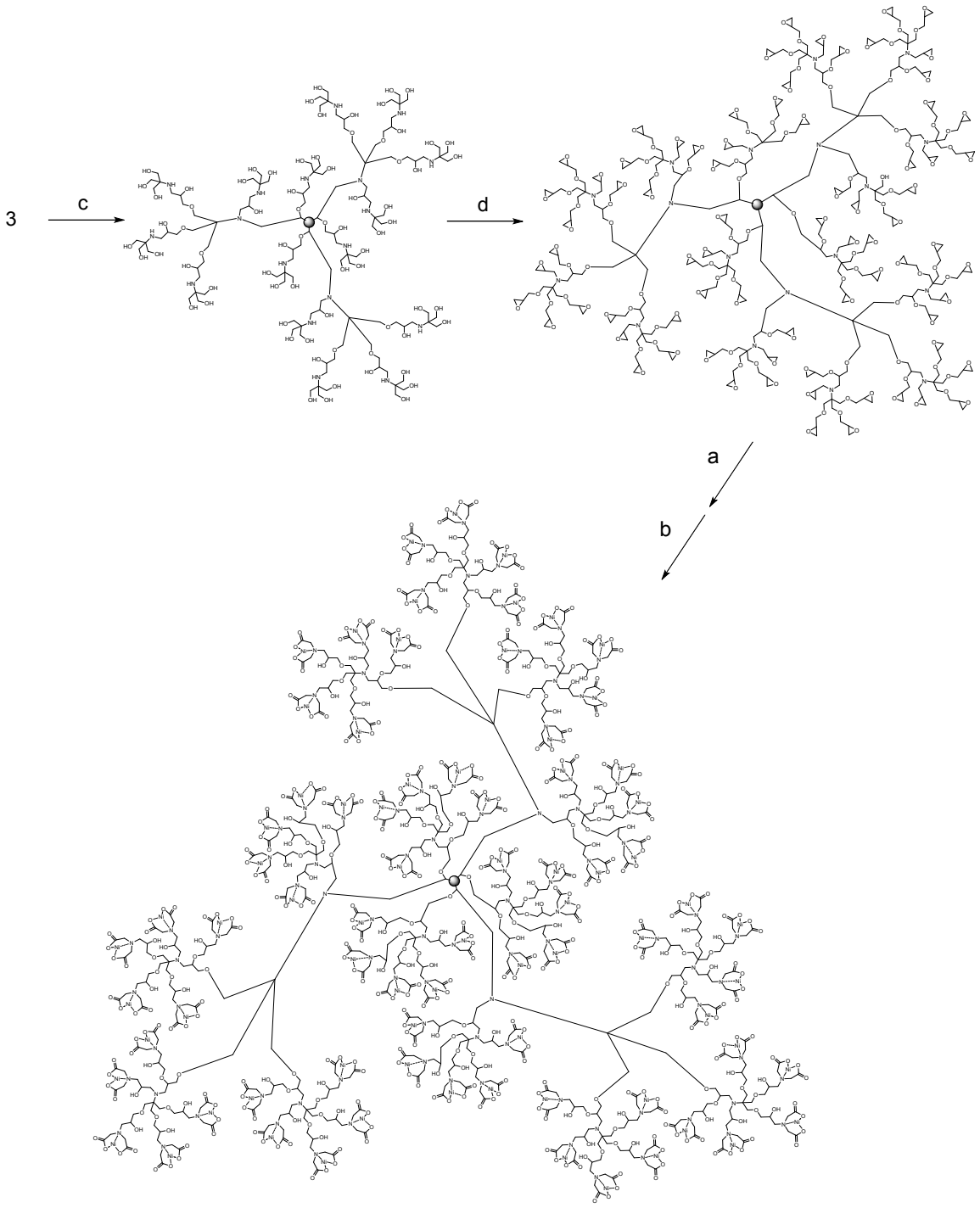
Series G1



1 2 3

G1

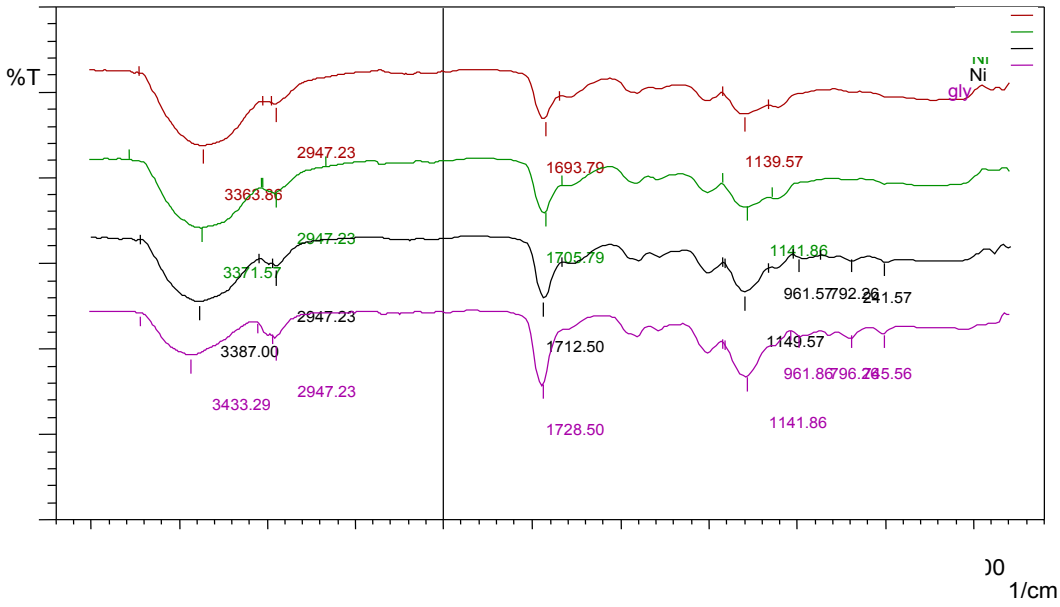
Series G2



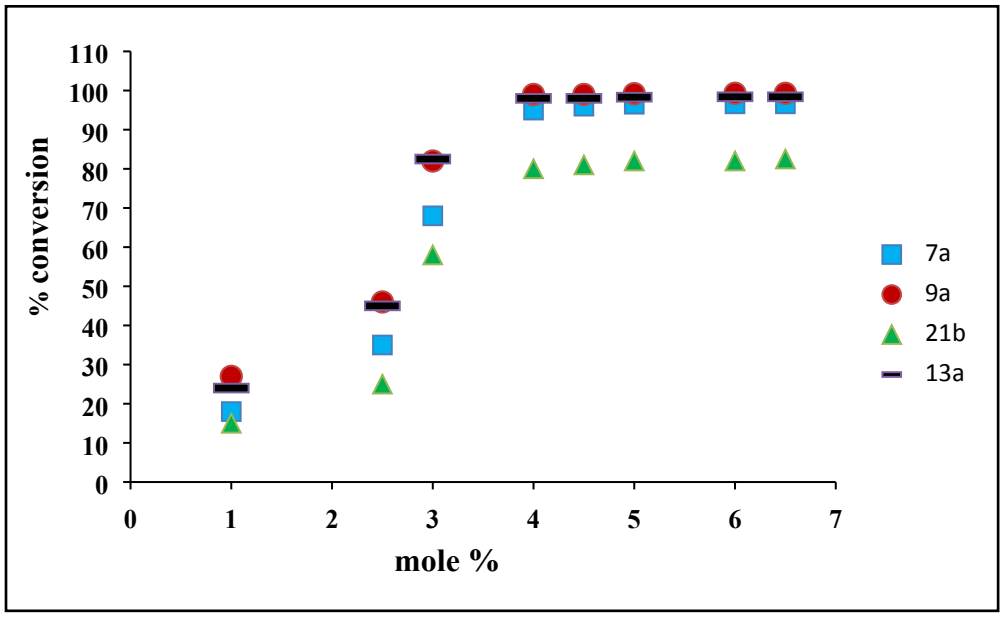
G2

Reagents**:** a =IDA, Na_2CO_3, 60^{o}C, 48h; b = 0.5M solution of $NiSO_4.7H_2O$; c = Tris, Na_2HPO_4, pH12, 60^{o}C, 48h; d = Epichlorohydrin, $NaBH_4$, RT, 48h

B) ATR-FTIR data of G0, G1 and G2 series of catalysts in comparison with starting material i.e. PMMA beads (Sepabeads EB-EP-400)



C) Conversion of amines to beta-amino alcohols (Table 2, entries 7a, 9a, 21b, 13a) at different mole% of Ni^{2+}-G2 sepabeads EB-SP-400



D) Comparison of performance of various catalysts for ring opening of epoxides with amines

Entry	Amines	Epoxides	Catalyst	Time (h)	Yield (%)	Ref
1	Aniline	Propylene oxide	$Y(NO_3)_3.6H_2O$	3	75	25
2	Aniline	Propylene oxide	$Sc(OTf)_3$	3	95	21
3	2-Fluoroaniline	Propylene oxide	$Y(NO_3)_3.6H_2O$	3	81	25
4	*o*-Toluidine	Propylene oxide	$Y(NO_3)_3.6H_2O$	3	90	25
5	*p*-Anisidine	Propylene oxide	$Y(NO_3)_3.6H_2O$	3	72	25
6	Aniline	Epichlorohydrin	$Y(NO_3)_3.6H_2O$	4	84	25
7	Aniline	Epichlorohydrin	NaY Zeolite	5	70	43
8	Aniline	Epichlorohydrin	Fe-MCM-41	2	98	26
9	2-Fluoroaniline	Epichlorohydrin	$Y(NO_3)_3.6H_2O$	4	82	25
10	Aniline	Styrene oxide	$B(C_6F_5)_3$	2-4	90	23
11	Aniline	Styrene oxide	$[Bmim]BF_4$	6.5	90	44
12	2-Fluoroaniline	Styrene oxide	$Y(NO_3)_3.6H_2O$	3	89	25
13	Amine	Epoxide	nano Fe3O4	20	70	47
14	Aniline	Epichlorohydrin	Ni^{+2}-G2 Sepabeads	0.5	97	This work
15	*p*-Aminophenol	Epichlorohydrin	Ni^{+2}-G2 Sepabeads	1.0	99	This Work

E) Calculation: Table.2 entries (1-21)

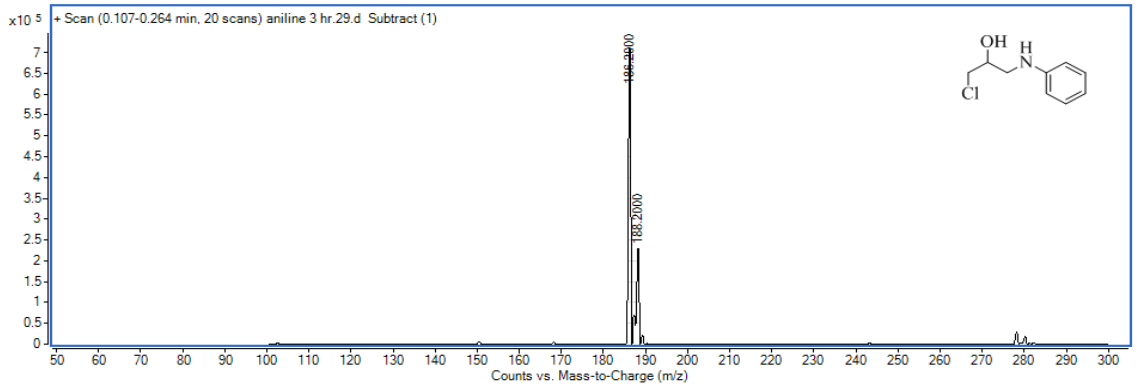
E factor = Total waste(mg)/product(mg)

Entry	Total waste(mg)	Product(mg)	E factor
1	12.0	88	0.136
2	5.0	95	0.052
3	7.0	93	0.075
4	4.0	96	0.041
5	6.0	94	0.063
6	2.0	98	0.020
7	4.0	96	0.041

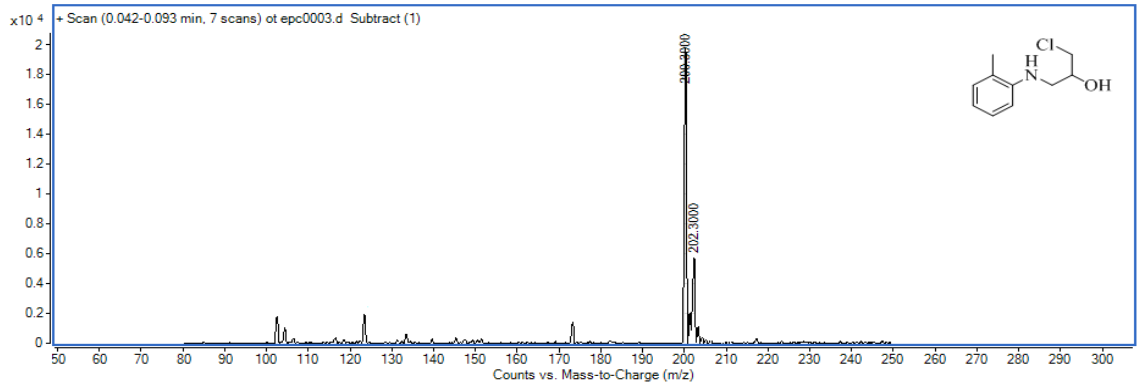
8	3..0	97	0.030
9	0.5	99.5	0.005
10	8.0	92	0.086
11	11.0	89	0.123
12	5.0	95	0.052
13	1.0	99	0.010
14	6.0	94	0.063
15	15.0	85	0.176
16	10.0	90	0.111
17	11.0	89	0.123
18	19.0	81	0.234
19	15.0	85	0.176
20	9.0	91	0.098
21	17.0	83	0.204

F) Mass Spectra of some selected pure product

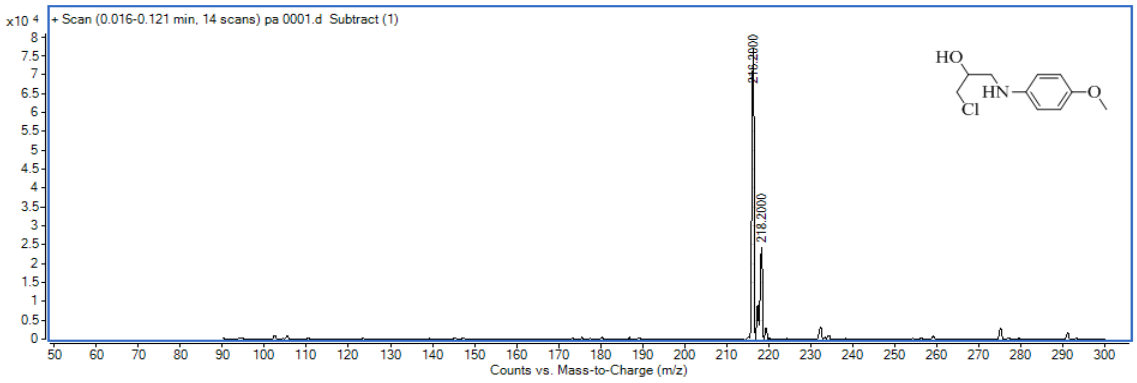
Aniline and epichlorohydrin



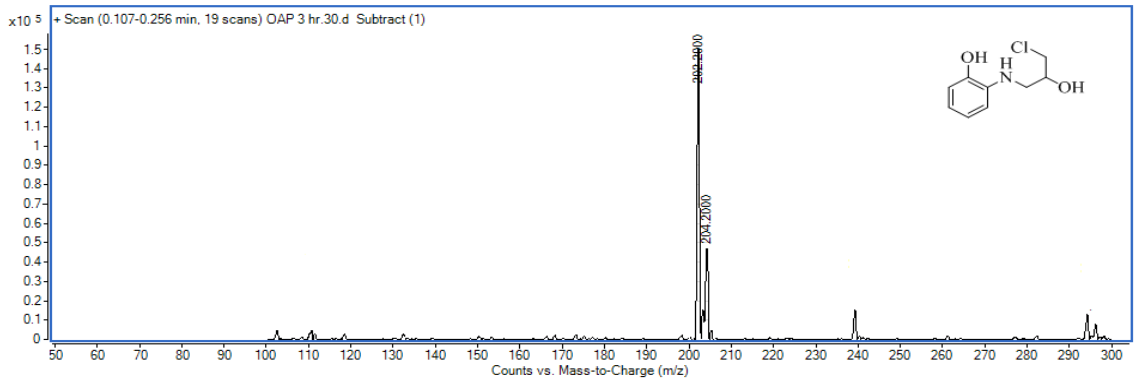
o- Toluidine and epichlorohydrin



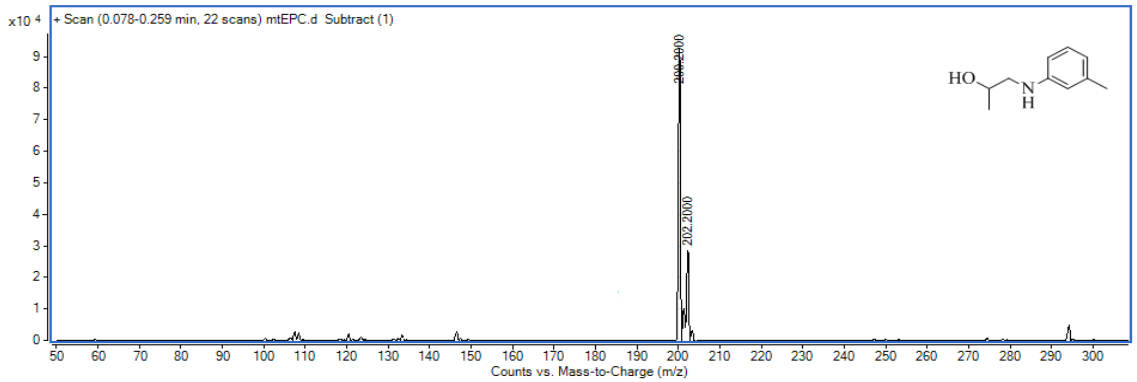
p-Anisidine and epichlorohydrin



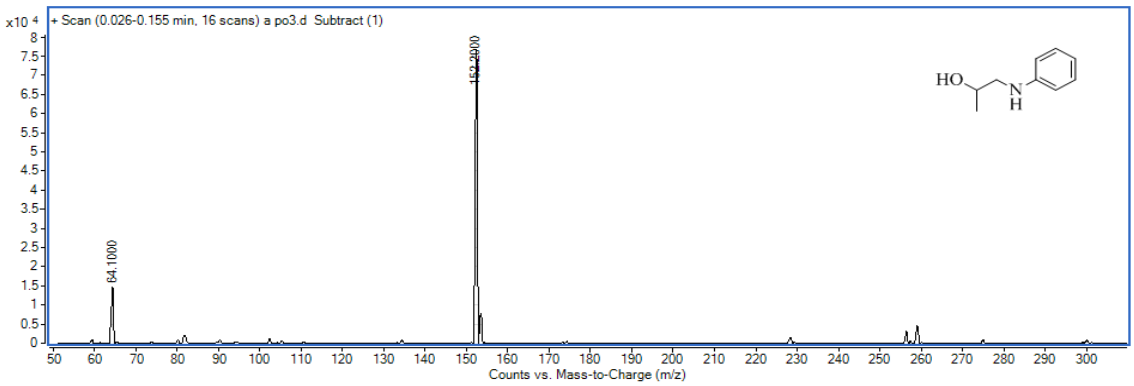
o- Amino phenol and epichlorohydrin



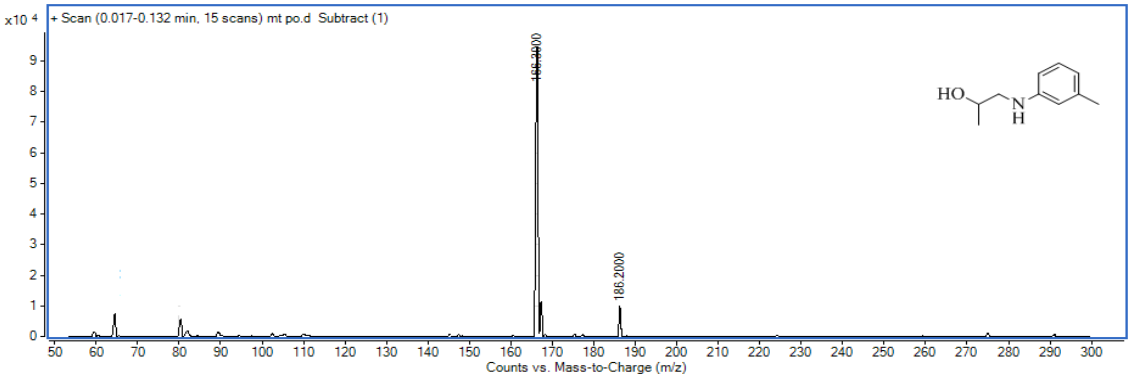
m- Toluidine and epichlorohydrin



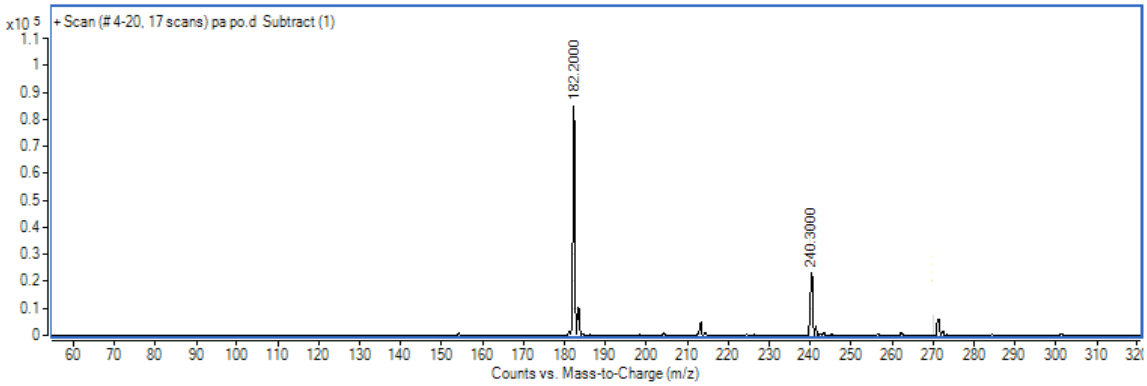
Aniline and propylene oxide



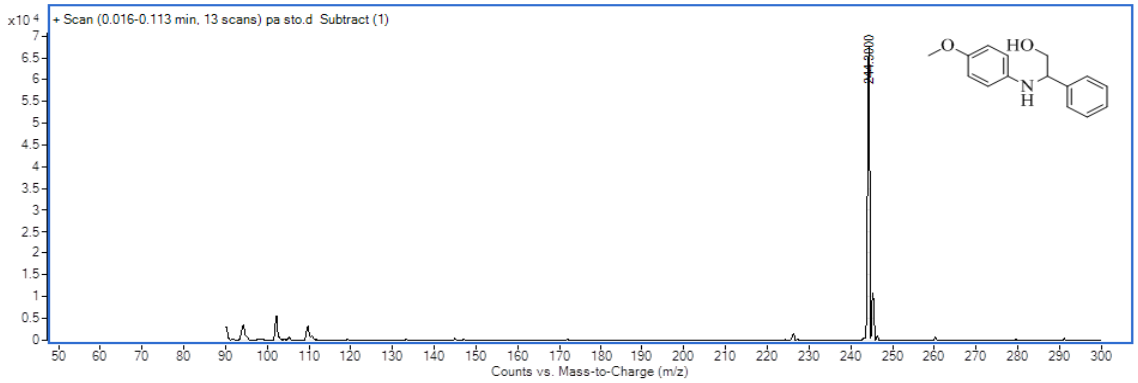
m-Toluidine and propylene oxide



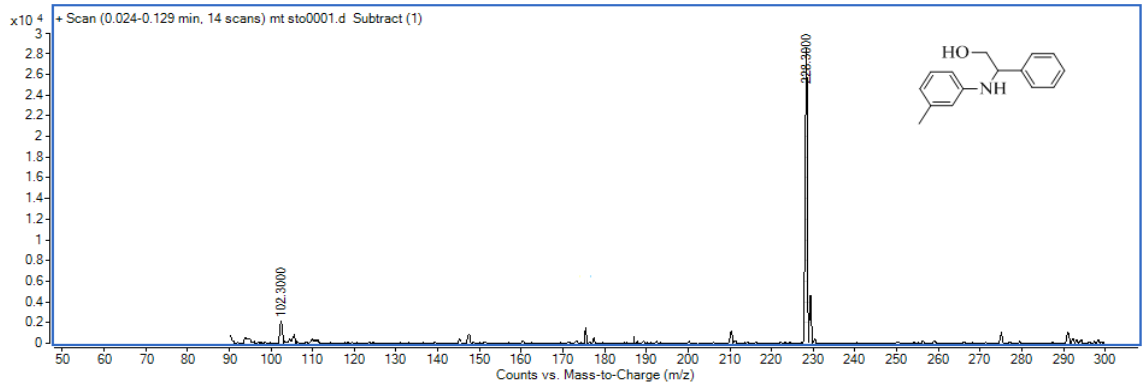
p-Anisidine and propylene oxide



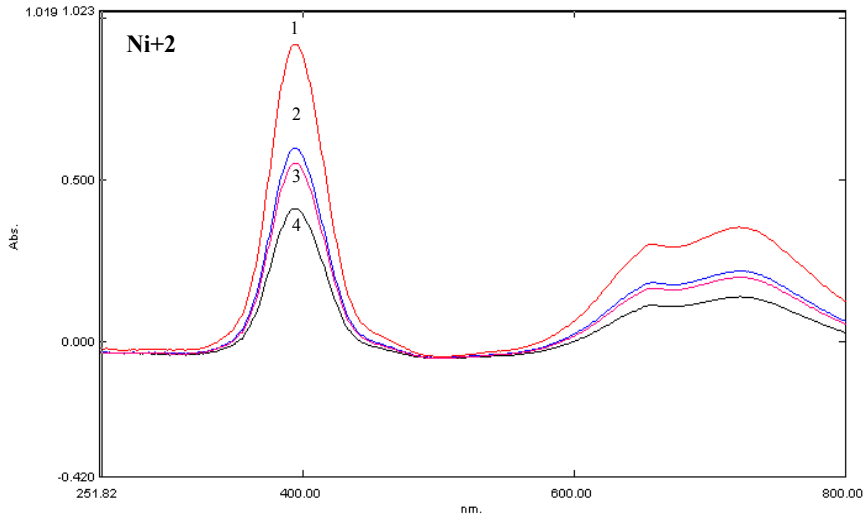
p-Anisidine and styrene oxide



m-Toluidine and styrene oxide



G) Representative UV-Vis Spectra of Ni^{+2} showing λ_{max} of 390 nm



1. 0.5M conc. metal load, 2. G0-Sepabeads, 3. G1-Sepabeads, 4. G2-Sepabeads

UV-vis spectra are employed to determine the concentration of metal ions before and after contact of metal ion solution with the mesoporous polymethacrylate beads. The absorbance of solution was determined at wavelength maxima (e.g. for Ni^{2+} as nickel sulfate solution is it 340 nm) and metal ion concentration was calculated by comparing absorbance with concentration of respective standards. A suitable dilution of the solution is performed to get the absorbance between 0.1 to 0.9 and is used while calculating the concentrations. Such calculations were used to determine the capacity of adsorption for all metals on G0, G1 and G2 series of dendritic side groups grafted Sepabeads respectively. The decrease in absorbance was observed after the contact of Ni^{+2}, Co^{+2}, Cu^{+2} and Fe^{+2} metals ions with G0, G1, and G2 series of dendritic side groups grafted Sepabeads.