APPENDIX

Chapter 2

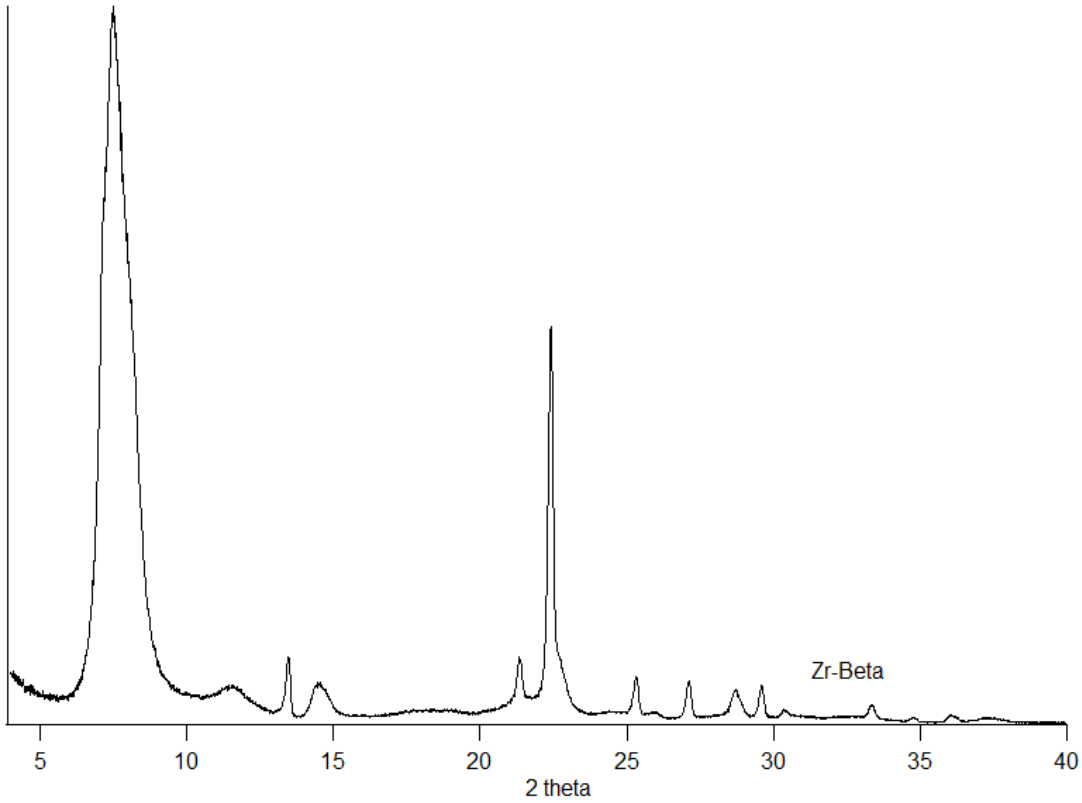

Fig. 2.1-A XRD pattern of Zr-Beta.

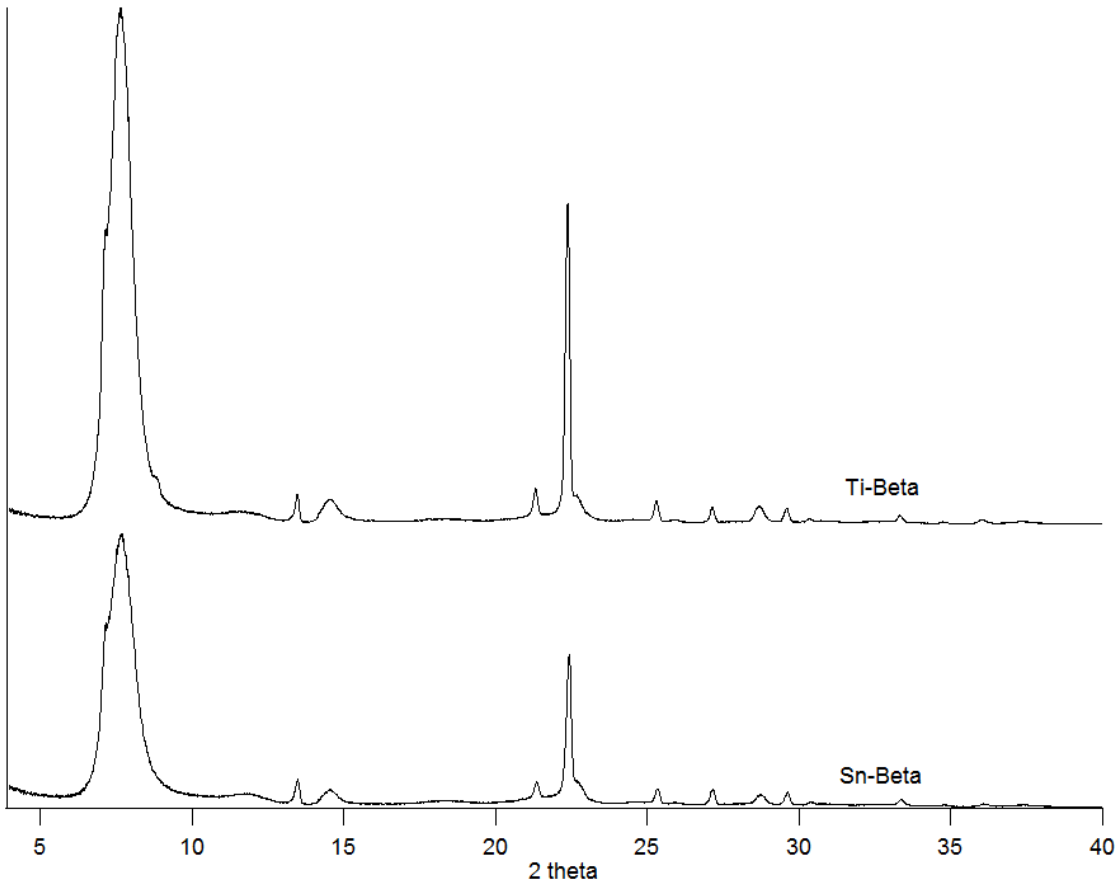

Fig. 2.2-A XRD patterns of Sn-Beta and Ti-Beta.

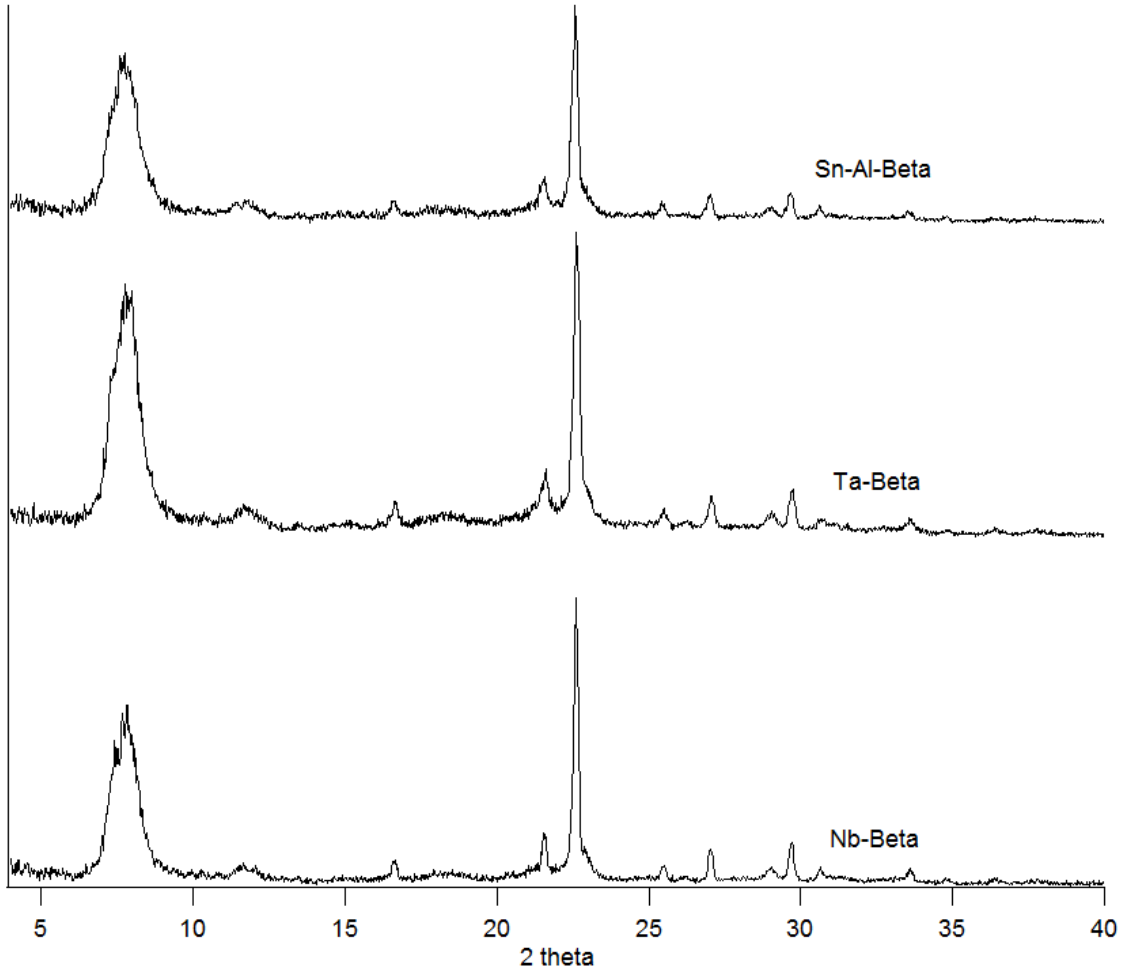

Fig. 2.3-A XRD patterns of Sn-Al-Beta, Ta-Beta, and Nb-Beta.

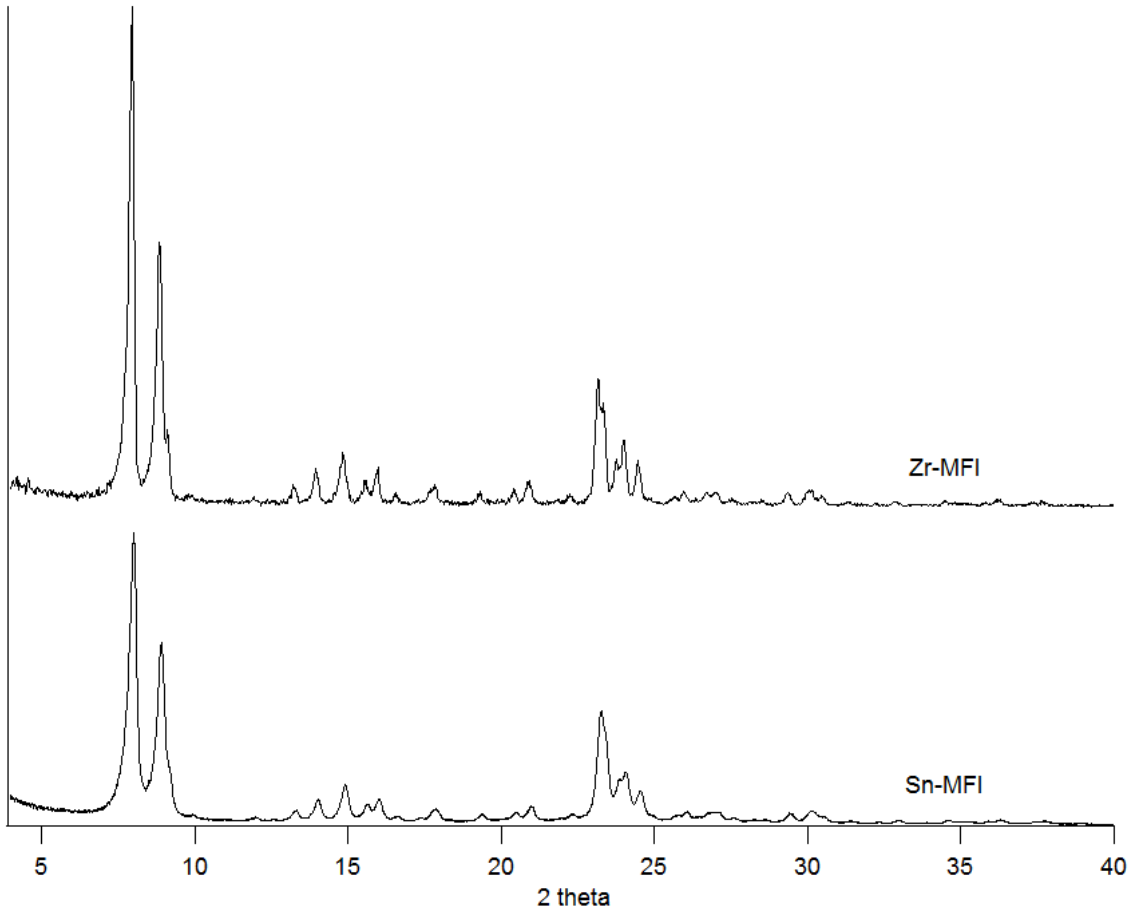

Fig. 2.4-A XRD patterns of Zr-MFI and Sn-MFI.

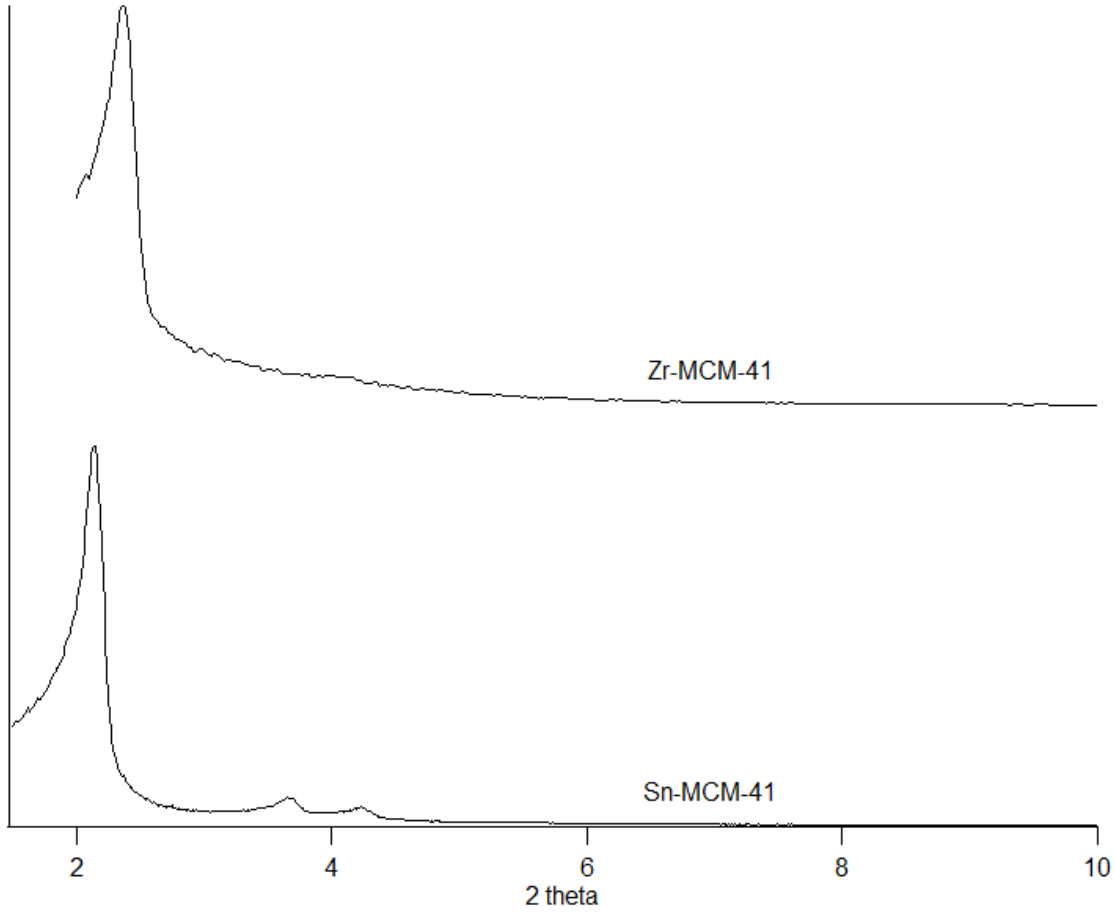

Fig. 2.5-A XRD patterns of Zr-MCM-41 and Sn-MCM-41.

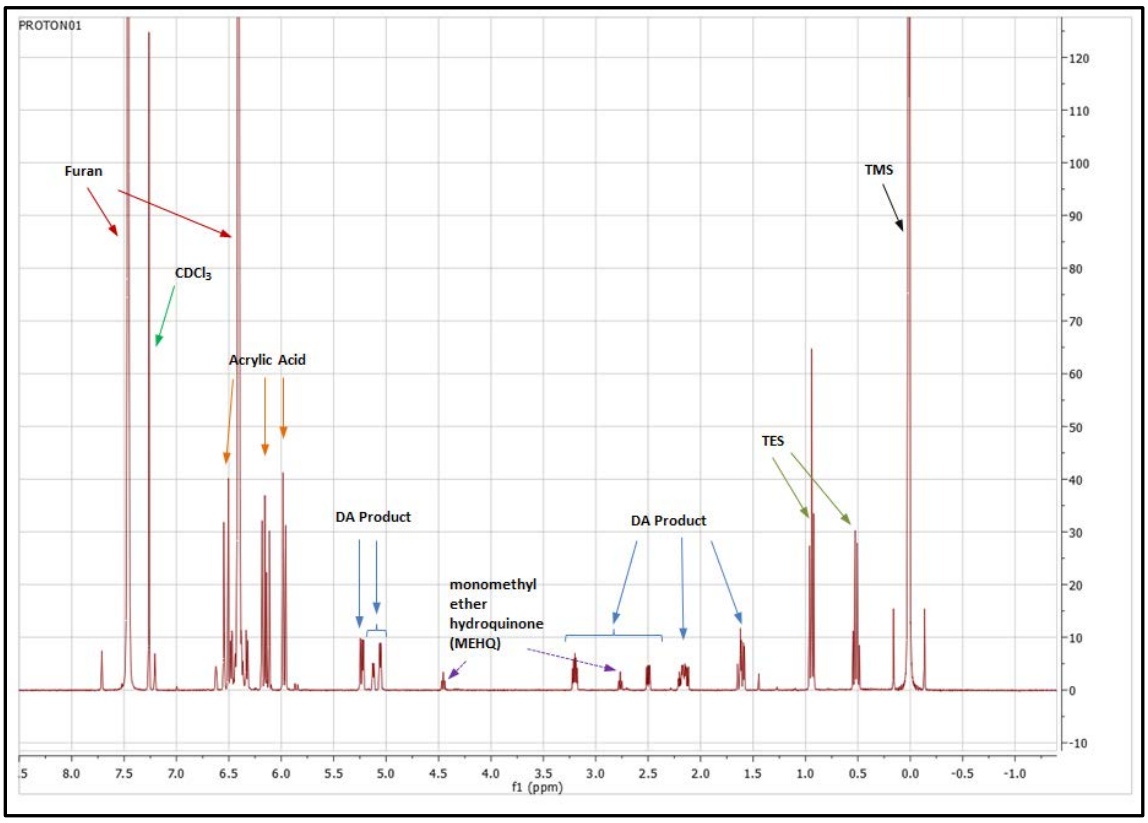

Fig. 2.6-A Example ^{1}H NMR spectrum taken in $CDCl_3$ of product solution of the Diels-Alder reaction between furan and acrylic acid with each of the peaks identified. The DA product, 7-oxa-bicyclo[2.2.1]hept-5-ene-2-carboxylic acid, was identified by using the product standard obtained from Matrix Scientific. All shifts are relative to TMS (tetramethylsilane). Monomethyl ether hydroquinone (MEHQ) was a stabilizer in the acrylic acid obtained from Sigma-Aldrich and no effort was made to remove the MEHQ prior to use.

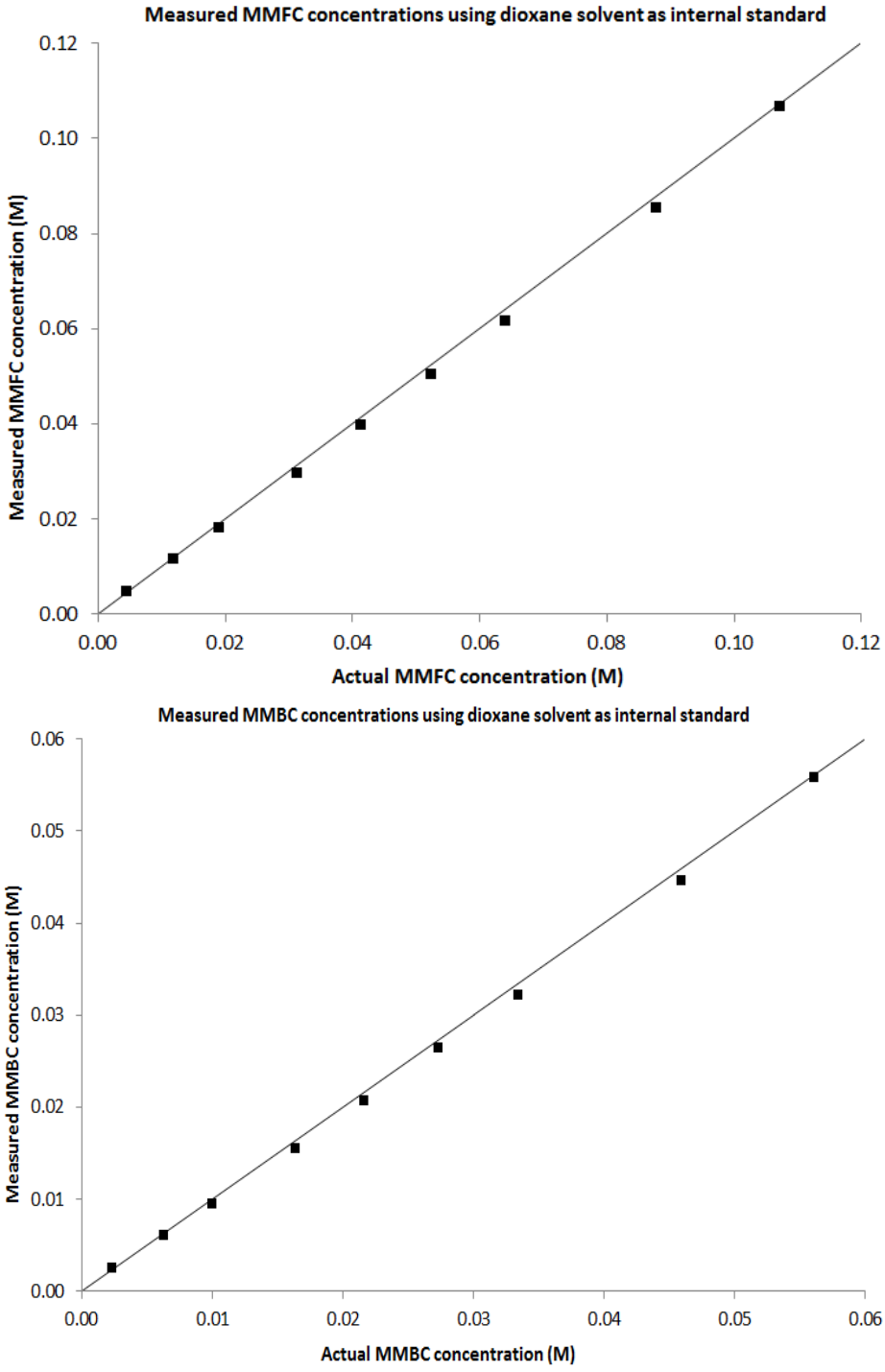

Fig. 2.7-A Measured MMFC and MMBC concentrations in dioxane by using the dioxane peak as internal standard for a series of solutions of known concentrations.

Table 2.1-A. Comparison of reaction results for the ethylene Diels-Alder-dehydration conversion of MMFC to MMBC using TES as external standard and the dioxane solvent as internal standard. Reaction conditions: 0.1 M MMFC solution in dioxane, 100 mg catalyst, 190^{o}C, 1000 psig C_2H_4.

Entry	Catalyst	Time (hr)	^{1}H NMR method	MMFC conversion (%)	MMBC yield (%)	MMBC selectivity (%)
1	Zr-Beta-185	4	TES external standard	21.6	16.3	75.5
			Dioxane internal standard	21.5	16.3	75.8
2	Zr-Beta-185	6	TES external standard	25.6	20.8	81.3
			Dioxane internal standard	27.6	19.9	72.1
3	Zr-Beta-185	12	TES external standard	42.1	32.2	76.5
			Dioxane internal standard	41.6	32.3	77.6
4	Zr-Beta-116	6	TES external standard	33.9	28.8	85.0
			Dioxane internal standard	32.2	30.0	93.2

Chapter 3

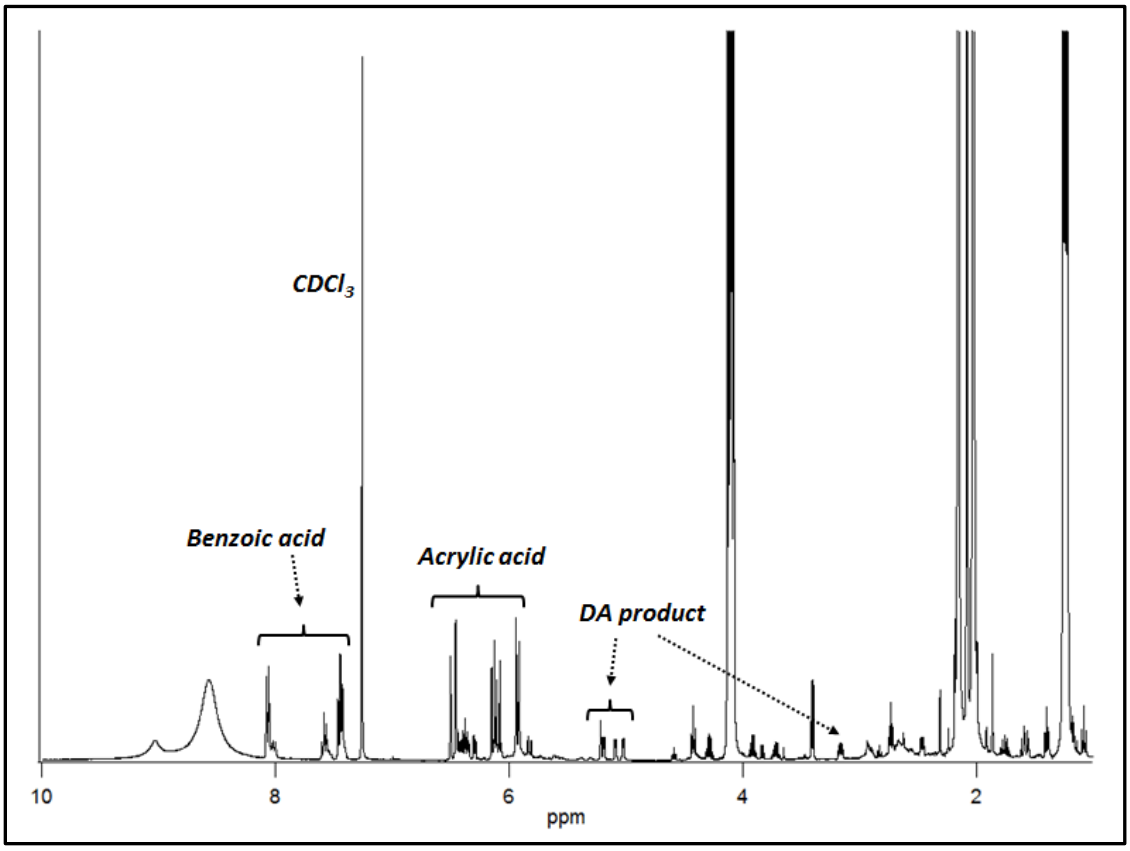

Fig. 3.1-A ^{1}H NMR spectrum (in $CDCl_3$) of the product obtained from the conversion of 7-oxa-bicyclo[2.2.1]hept-5-ene-2-carboxylic acid to benzoic acid using concentrated sulfuric acid at 0^{o}C. The benzoic acid product, acrylic acid (formed by the retro-Diels-Alder reaction), and unreacted 7-oxa-bicyclo[2.2.1]hept-5-ene-2-carboxylic acid are identified.

Chapter 4

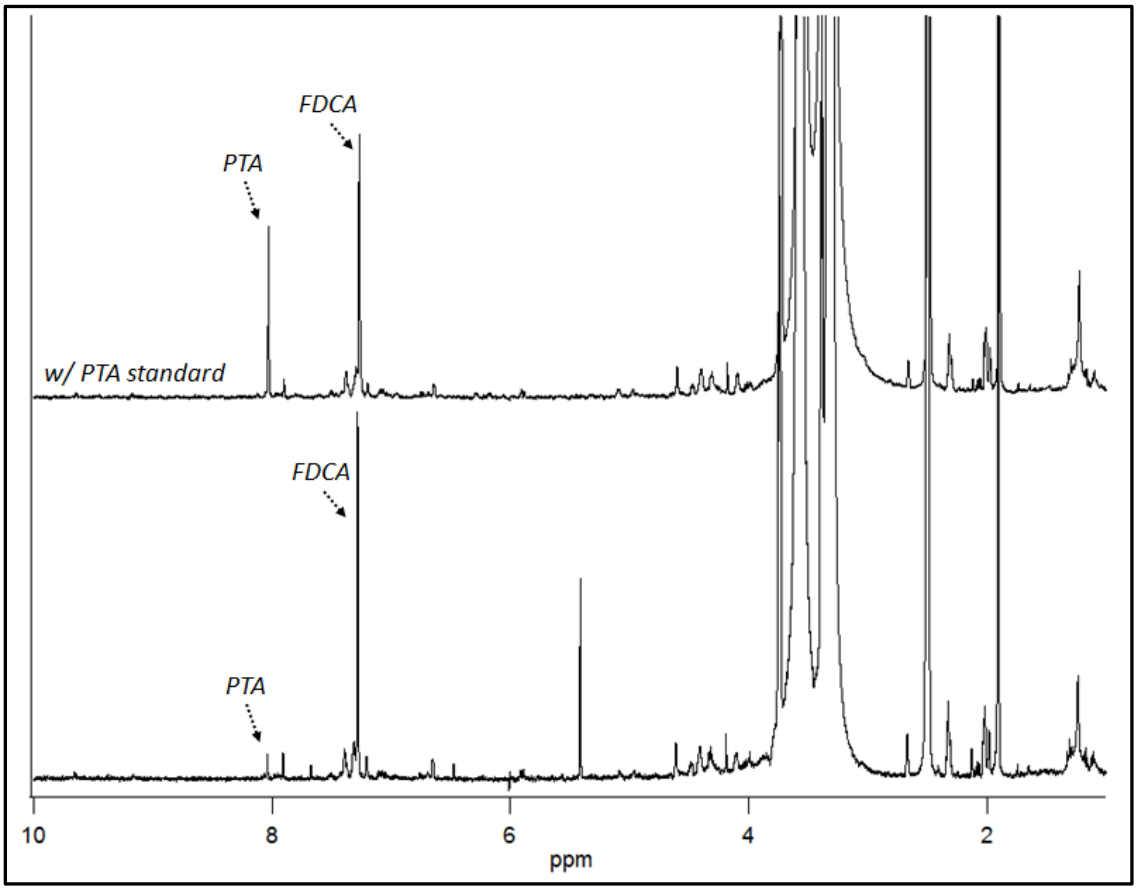

Fig. 4.1-A Diels-Alder-dehydration of FDCA and ethylene to PTA. ^{1}H NMR (in DMSO-d_6) of crude product solution (bottom) and product solution spiked with PTA product standard (top). Dioxane solvent shows up at 3-4 ppm. Reaction conditions: 0.1 M FDCA in dioxane, 200 mg Sn-Beta, 250^{o}C, 1000 psig C_2H_4, 14 hours.

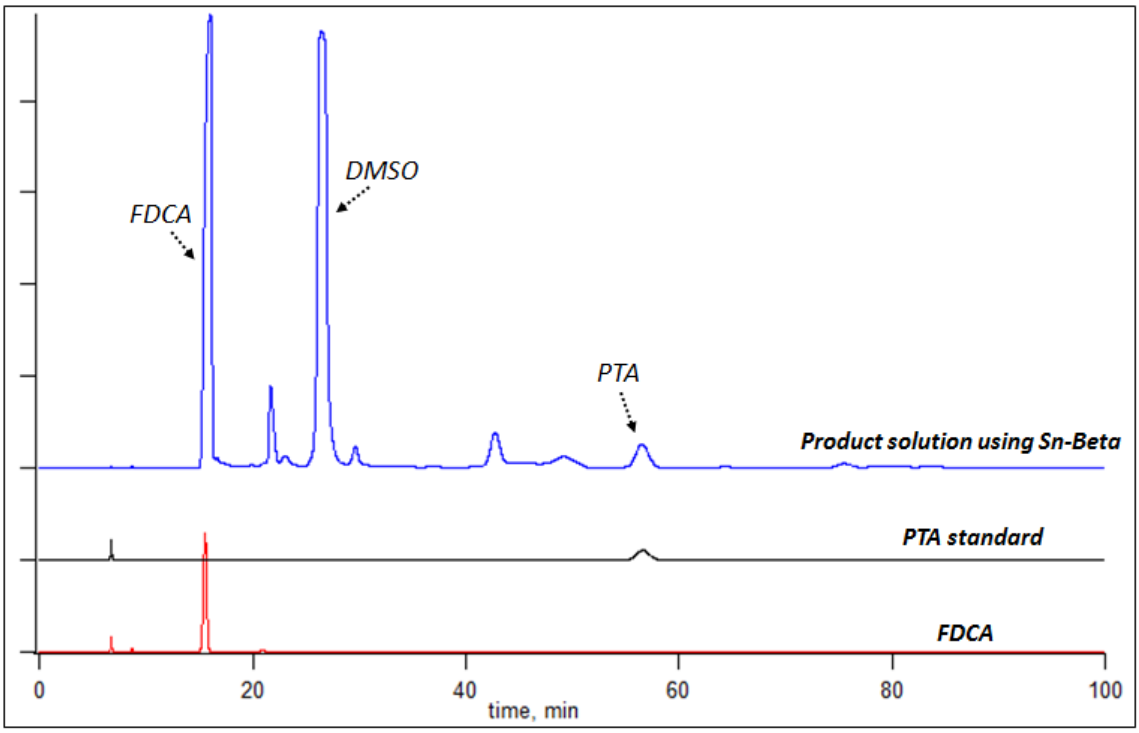

Fig. 4.2-A Diels-Alder-dehydration of FDCA and ethylene to PTA. HPLC chromatograms of reaction product solution using Sn-Beta catalyst (top), PTA product standard (middle), and FDCA reactant (bottom). DMSO is present in the product solution since DMSO was used to solubilize all of the FDCA and PTA while recovering from reactor. Reaction conditions: 1 M FDCA in dioxane, 200 mg Sn-Beta, 225^{o}C, 1000 psig C_2H_4, 16 hours.

Chapter 5

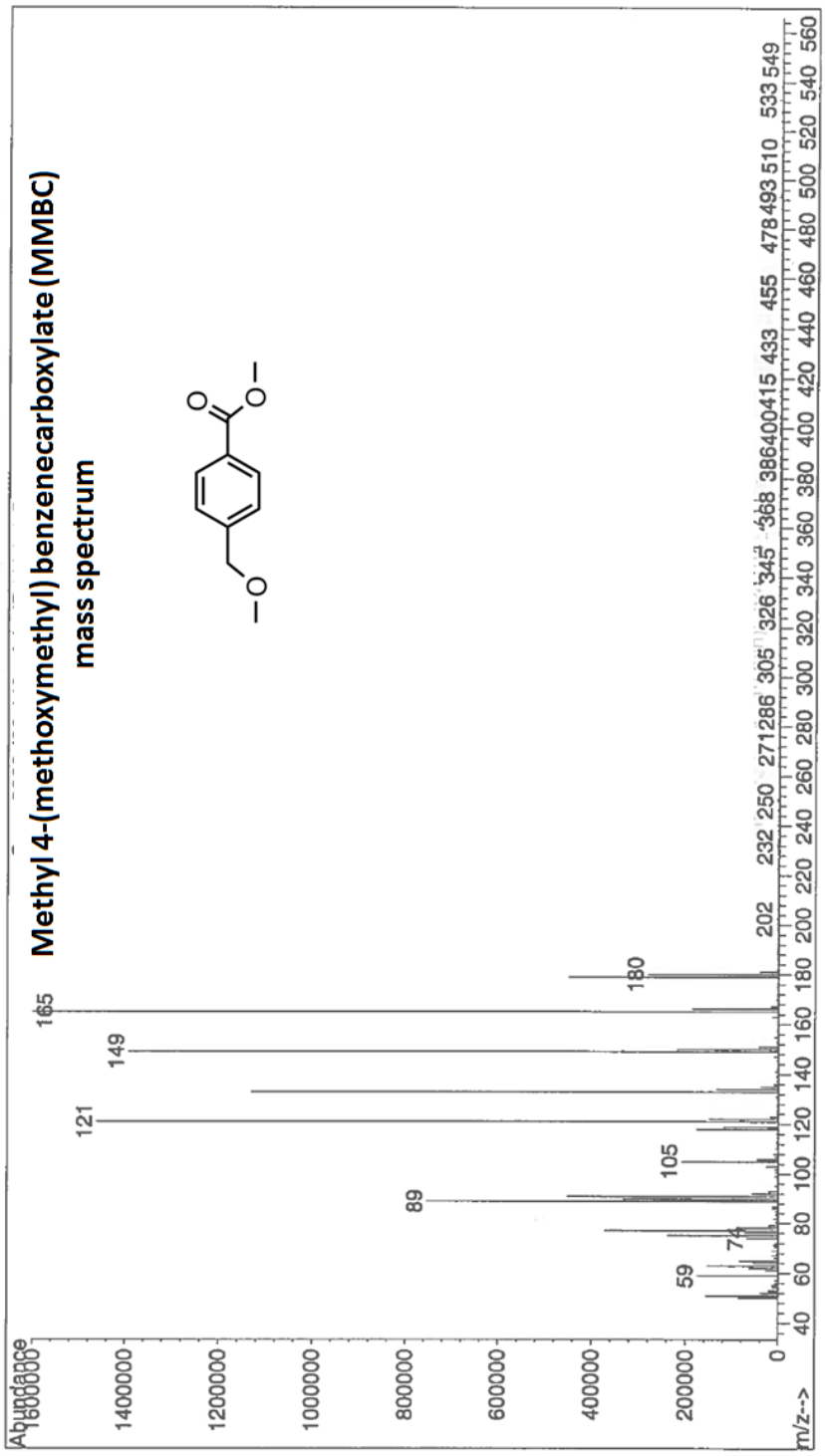

Fig. 5.1-A EI mass spectrum of methyl 4-(methoxymethyl) benzenecarboxylate (MMBC) product in the Diels-Alder-dehydration of MMFC and ethylene.

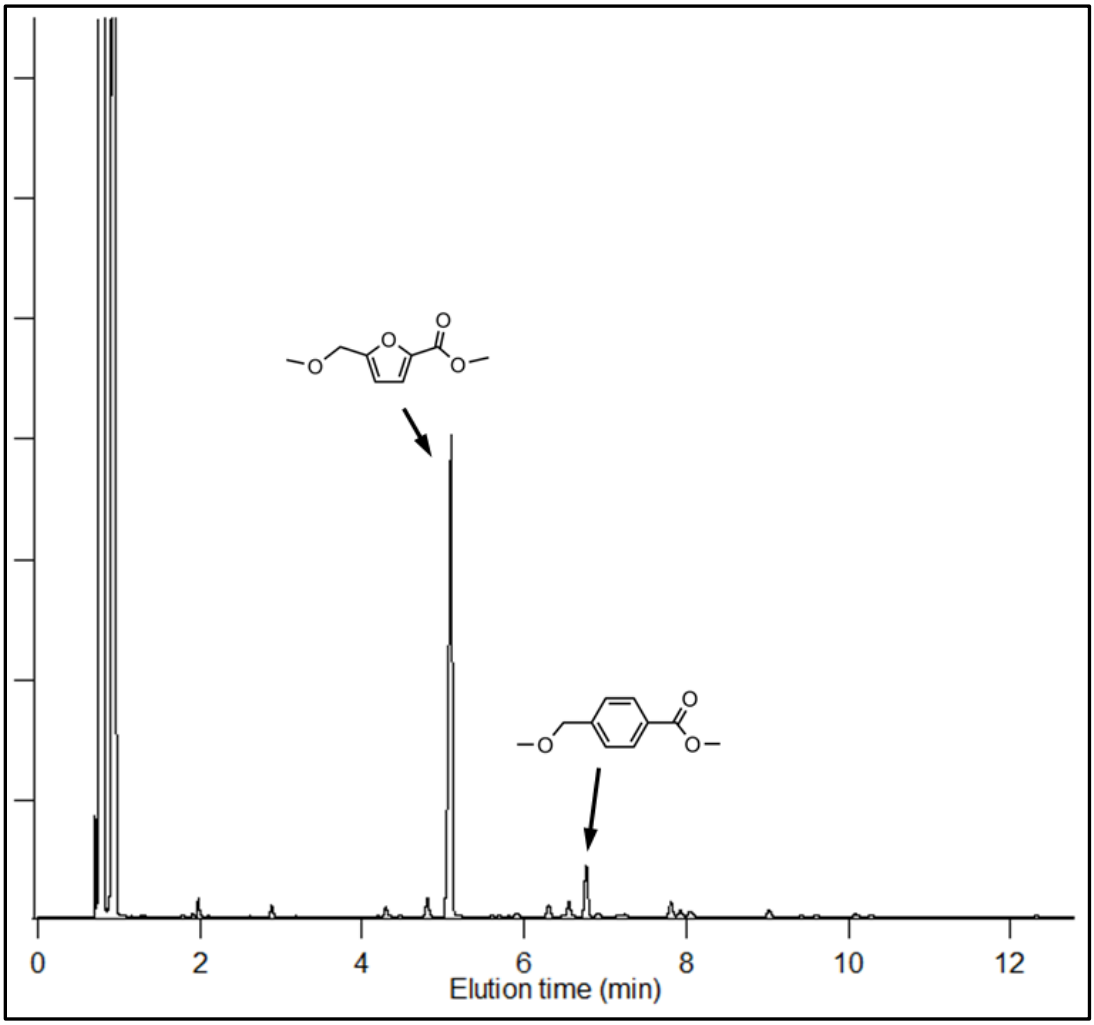

Fig. 5.2-A GC-FID chromatogram of product solution for ethylene Diels-Alder-dehydration of MMFC using high MMFC concentrations. Reaction conditions: 10 wt % MMFC in dioxane, 190^{o}C, 6 hr, 1000 psig C_2H_4.

Chapter 6

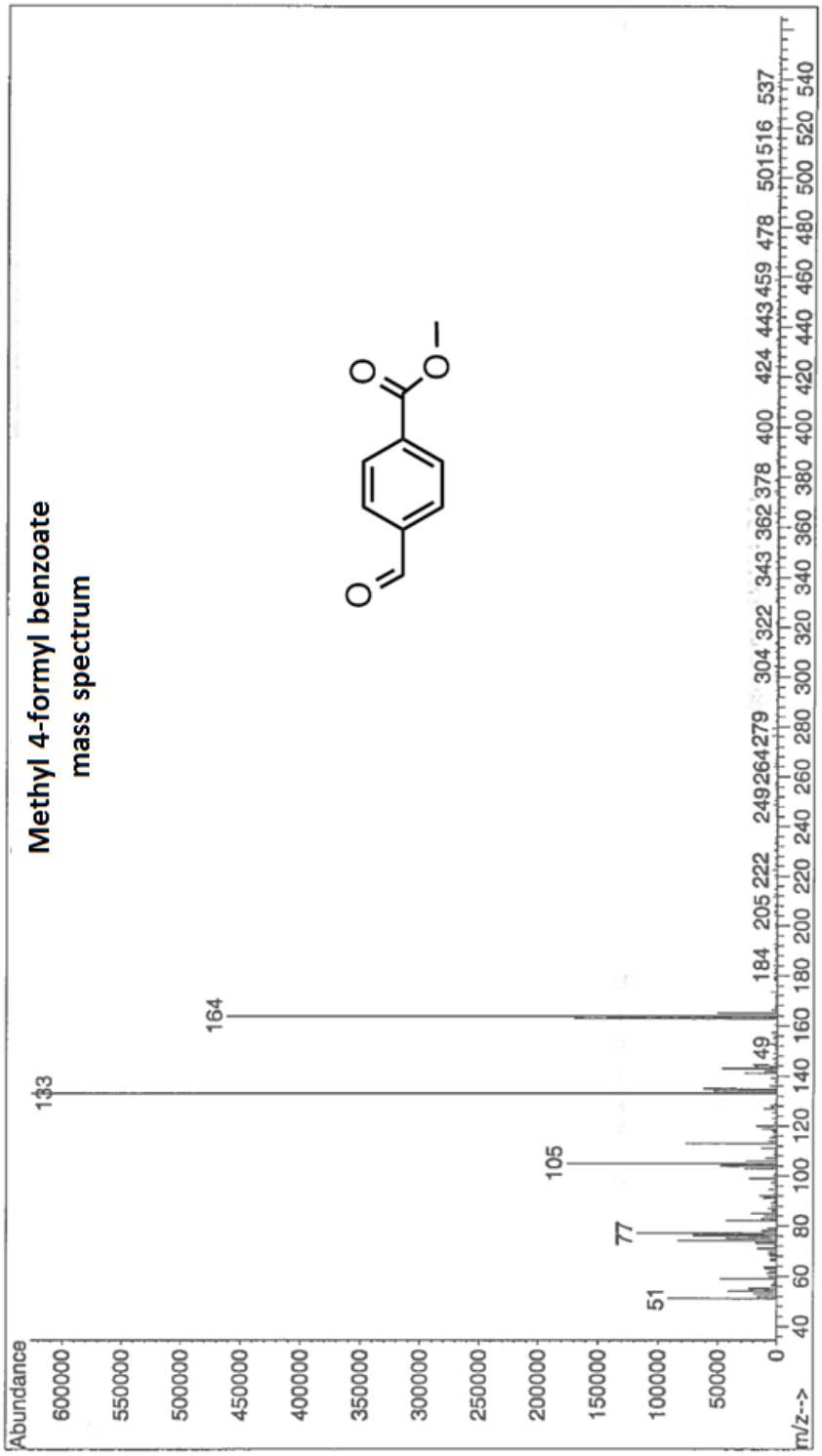

Fig. 6.1-A EI mass spectrum of methyl 4-formyl benzoate in Fig. 6.11.

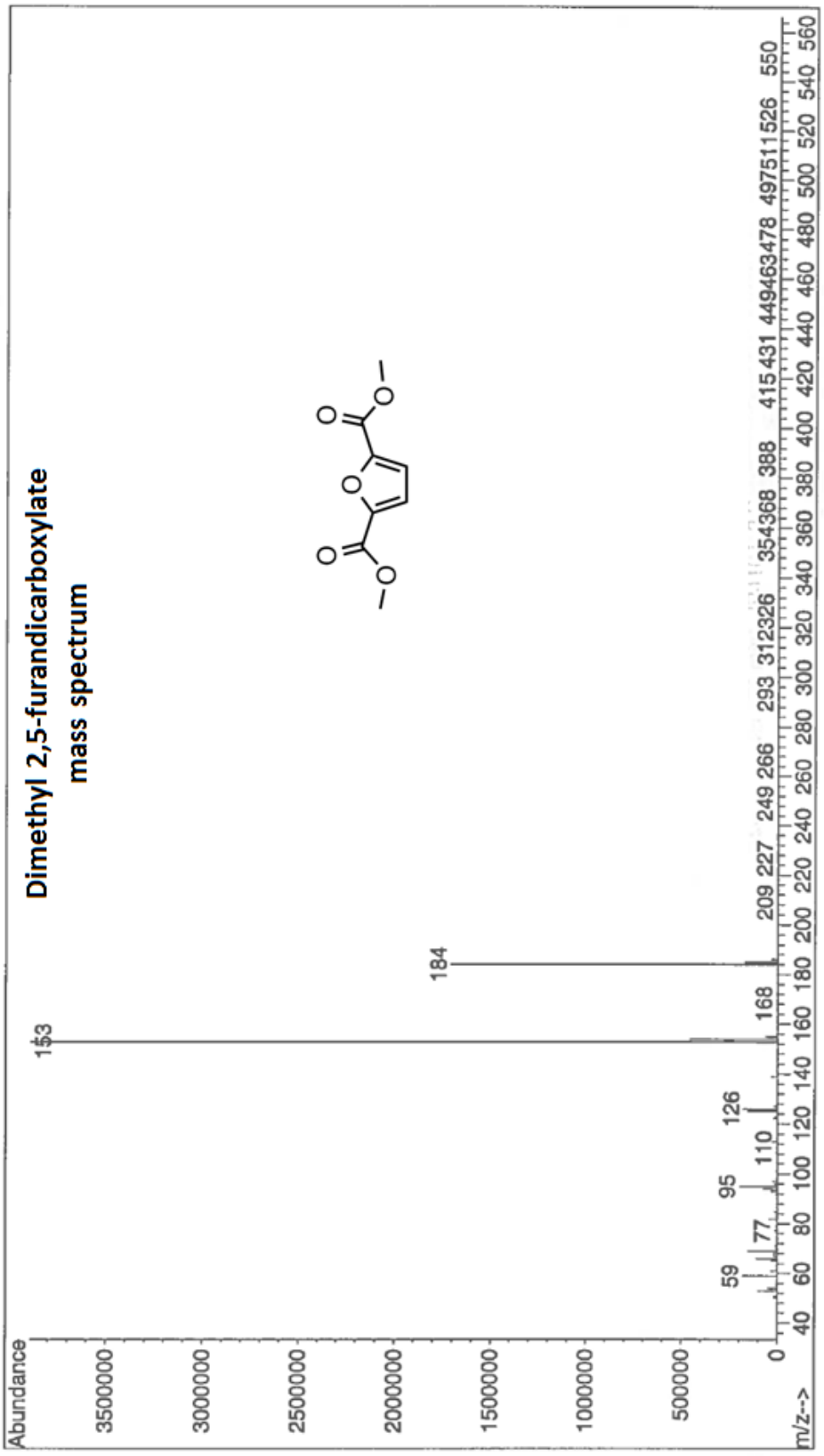

Fig. 6.2-A EI mass spectrum of dimethyl 2,5-furandicarboxylate in Fig. 6.11.

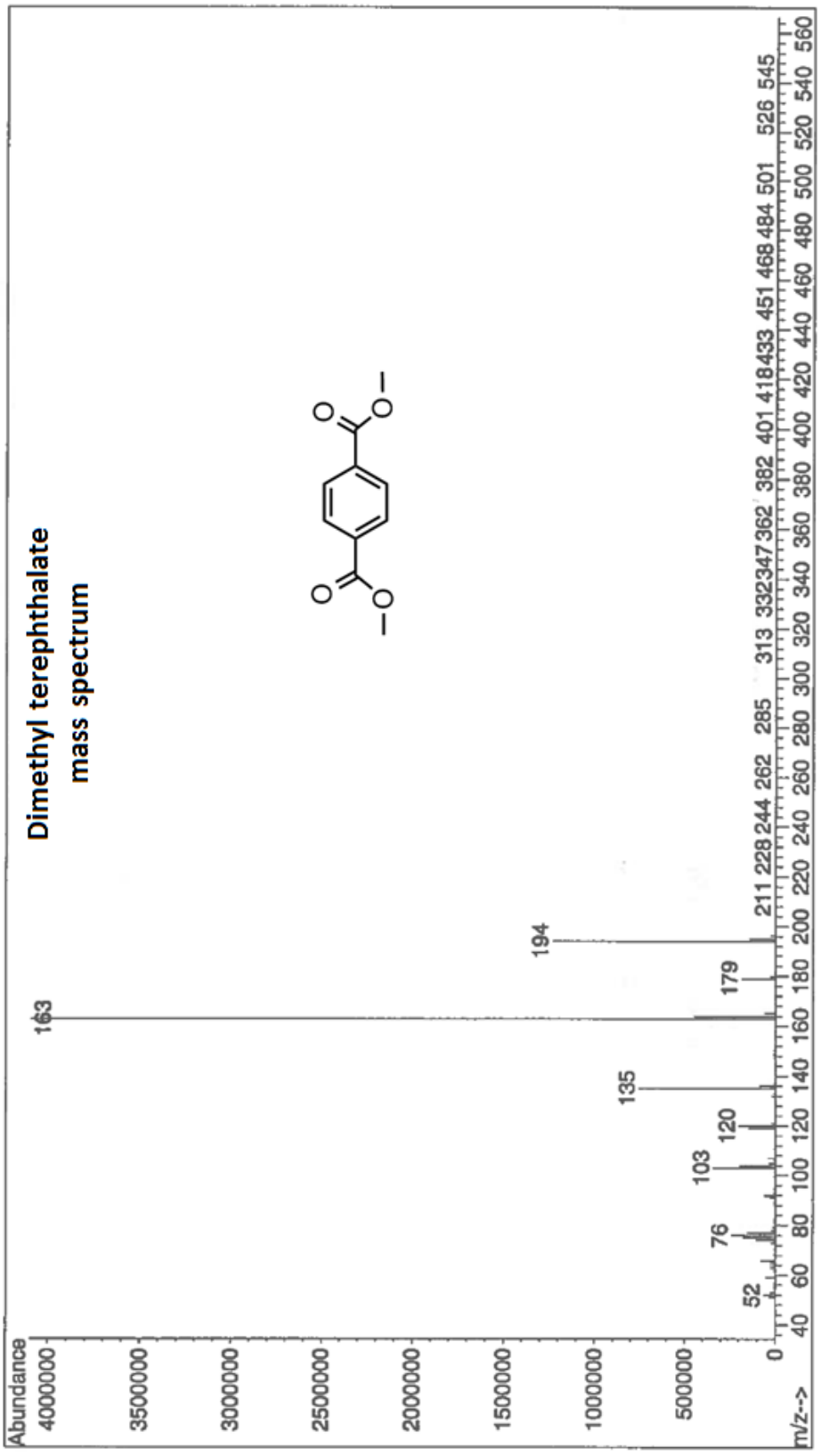

Fig. 6.3-A EI mass spectrum of dimethyl terephthalate in Fig. 6.11.

Chapter 7

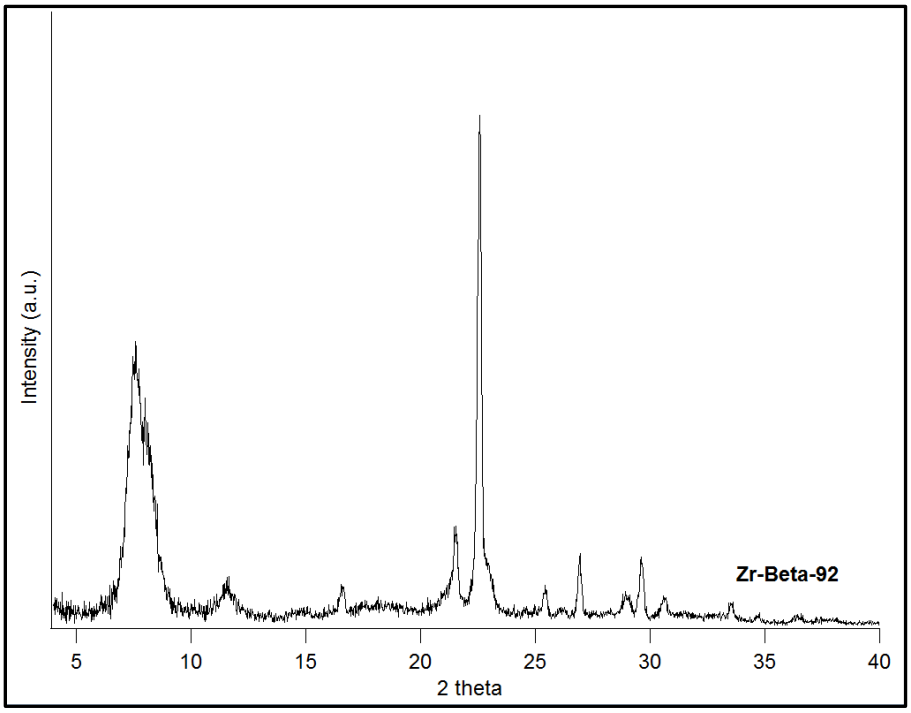

Fig. 7.1-A XRD pattern of Zr-Beta-92.

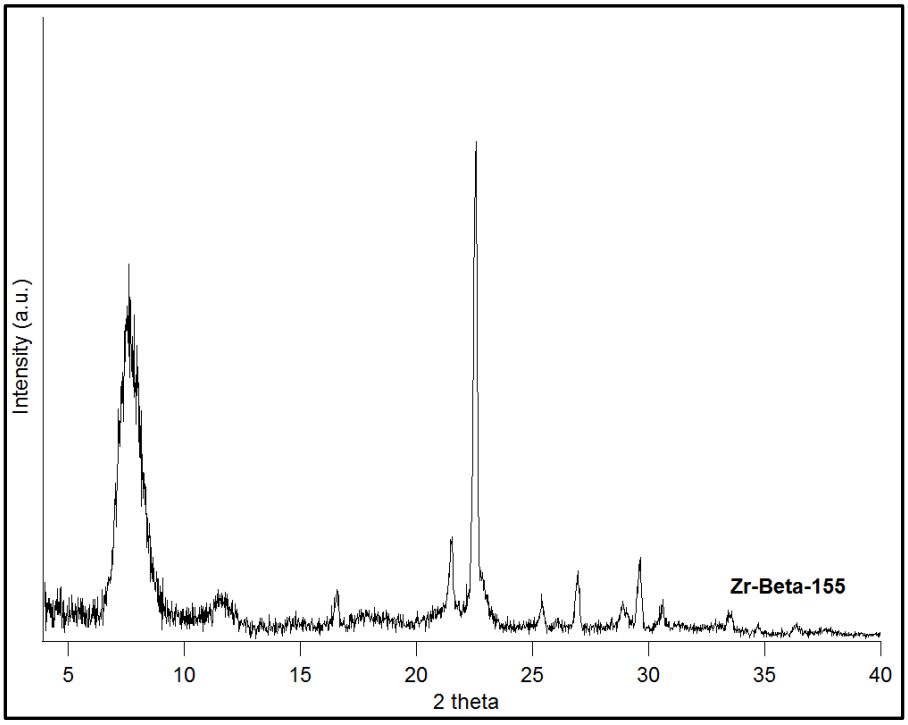

Fig. 7.2-A XRD pattern of Zr-Beta-155.

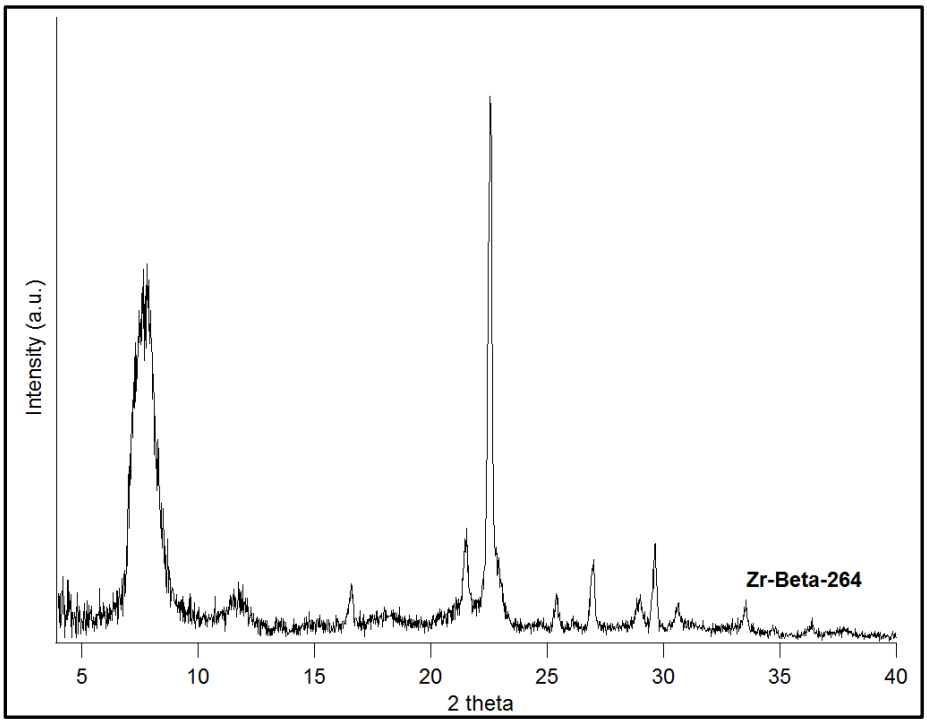

Fig. 7.3-A XRD pattern of Zr-Beta-264.

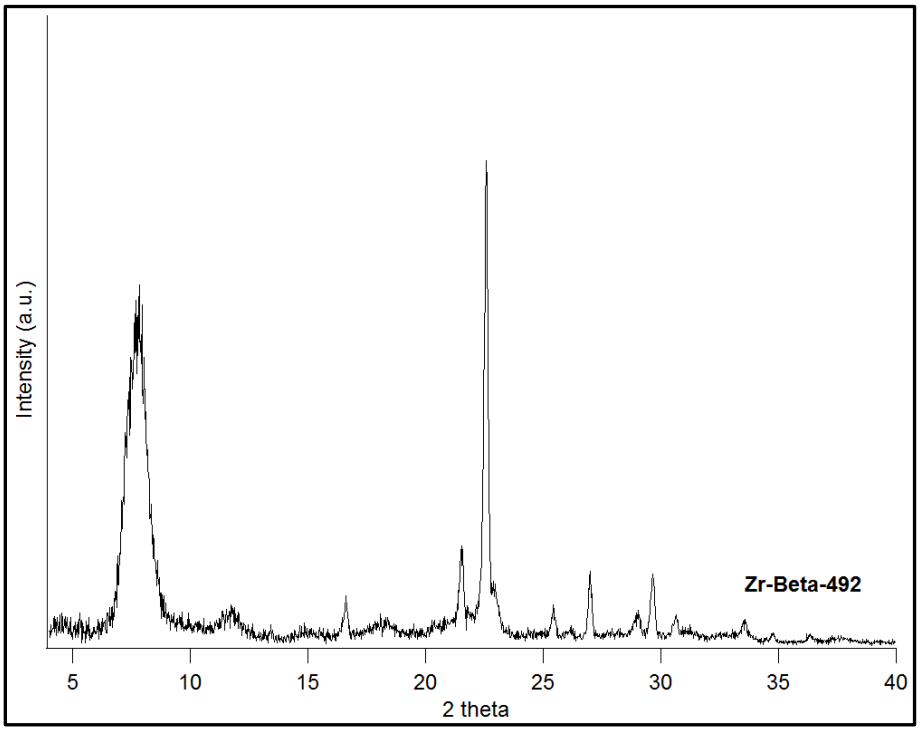

Fig. 7.4-A XRD pattern of Zr-Beta-492.

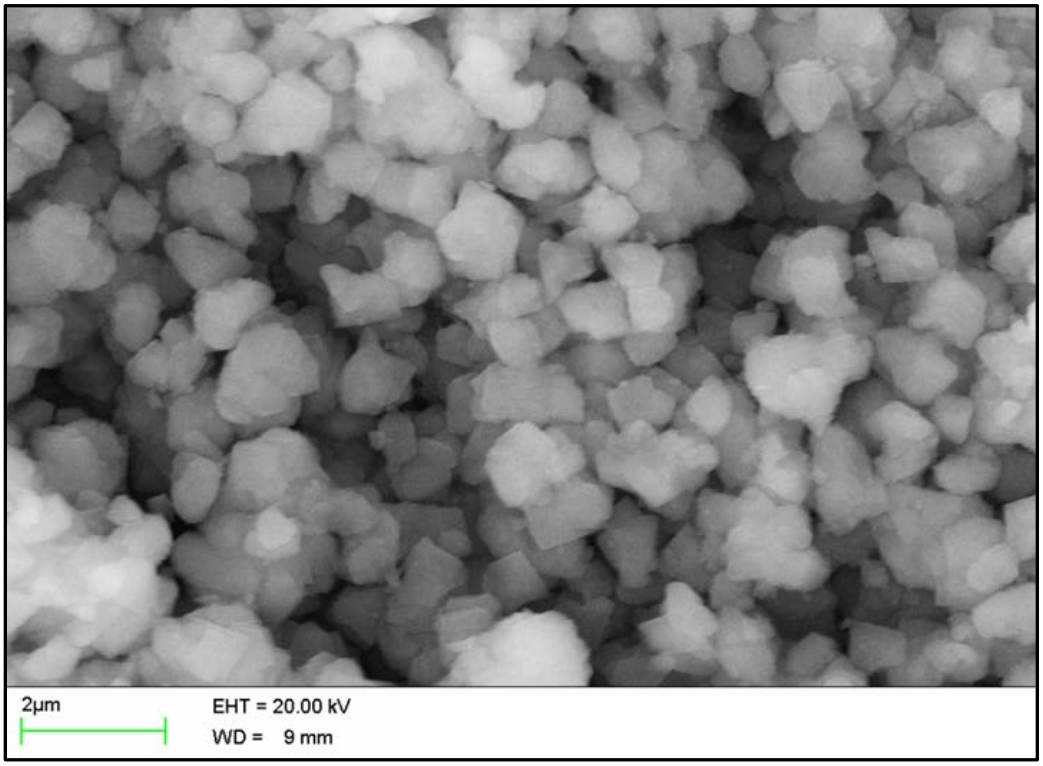

Fig. 7.5-A SEM image of Zr-Beta-92.

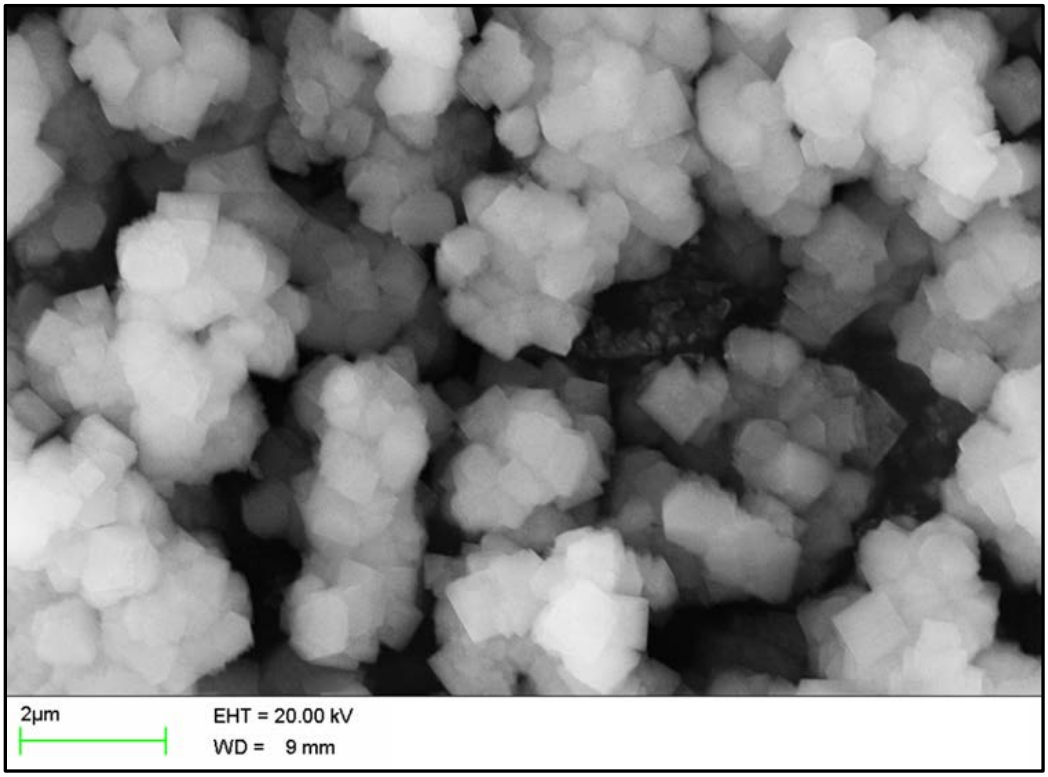

Fig. 7.6-A SEM image of Zr-Beta-155.

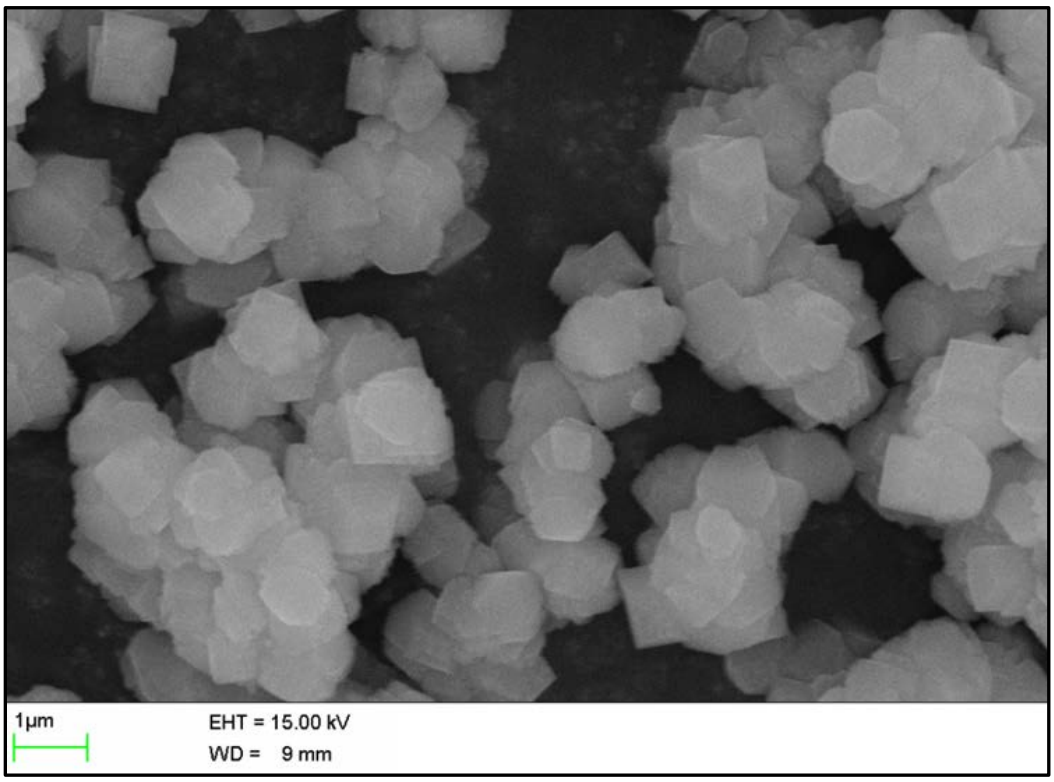

Fig. 7.7-A SEM image of Zr-Beta-264.

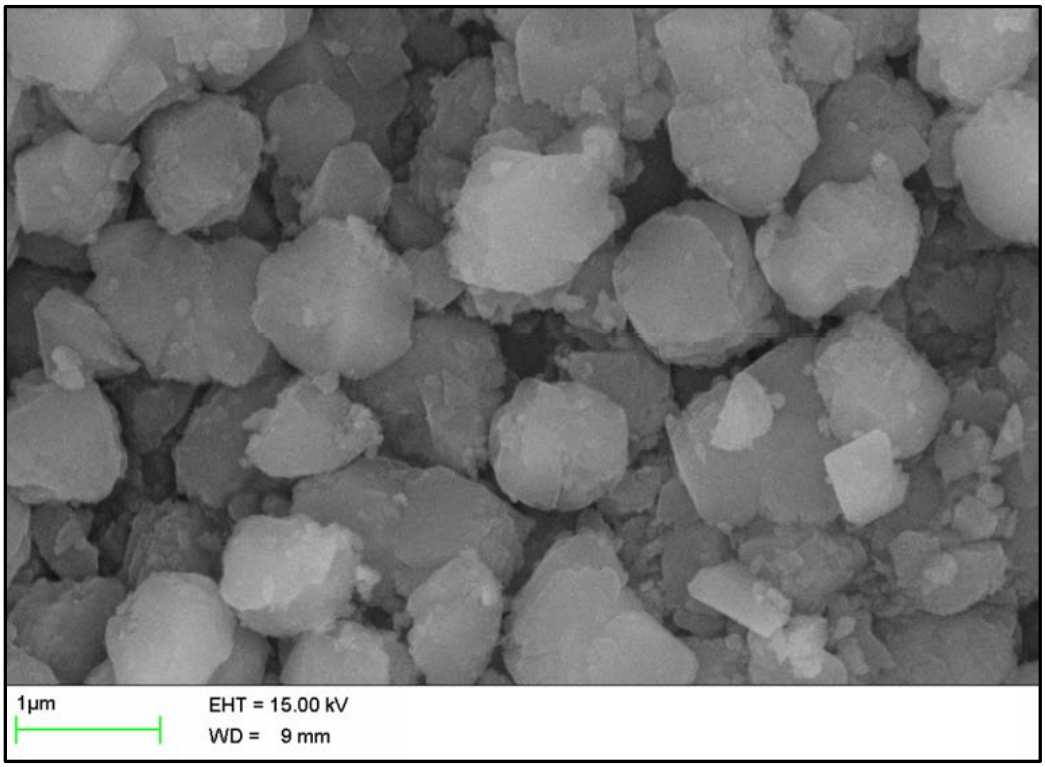

Fig. 7.8-A SEM image of Zr-Beta-492.