Electronic Supplementary Material (ESI) for Catalysis Science & Technology
This journal is © The Royal Society of Chemistry 2012

Supplementary Section

Synthesis and Characterization studies of γ-Al_2O_3 -supported, V-Zr mixed oxide catalysts prepared from $OV(OEt)_3$ and $Zr(O^nBu)_4$

Arindom Saha†*, Darrell P. Eyman
Department of Chemistry, University of Iowa, Iowa City, IA 52242-1294, USA.
Email: aus33@psu.edu ; Tel: 319-621-1452.

2. Experimental Section (contd.)

2.1 Sample synthesis and nomenclature (contd.)

Using a micropipette, measured amounts of metal alkoxide precursors of vanadium and zirconium oxides were placed in two-necked round bottom flasks inside an inert atmosphere glove box. The flasks were then made air-tight by covering them with rubber septa. After removal from the glove box the flasks were attached to a Schlenk line and the solvent (THF) was added to the precursors using an air-tight hypodermic syringe. The solutions were then refluxed under inert nitrogen atmosphere for at least 2 h. A measured amount of γ-Al_2O_3 pellets was then added to the mixed metal alkoxide solution under the protection of a stream of nitrogen to prevent any moisture from getting into the reaction vessel. Depending on the study being pursued the co-grafting solutions were maintained at certain constant temperatures for a given amount of time. The excess solvent along with the unreacted alkoxides were then discarded and the solid products left behind were thoroughly washed with solvent (THF) until no further metal oxide formation was observed on addition of water to the rejected washings. Following this, the solid products were hydrolyzed with water vapor in a N_2 stream while holding them at a constant temperature of 60°C in an oil bath for 6 h. The

This journal is © The Royal Society of Chemistry 2012

samples were then calcined overnight at 500°C to get the final co-grafted V- Zr mixed oxide catalysts.

Table S1. Percentage of concentration of different species present in samples synthesized from $OV(OEt)_3$ and $Zr(O^nPr)_4$ on calcination at 500°C.

Sample	Calcination Temperature (° C)	% Concentration		
		V^{5+}	Zr^{4+}	Al^{3+}
Time	500	10.68	34.91	54.41
Feed	500	3.47	7.98	88.55
Conc.	500	0.65	0.71	98.64

This journal is © The Royal Society of Chemistry 2012

Table S2. Percentage concentration of different species present in samples synthesized from $OV(OEt)_3$ and $Zr(O^nPr)_4$ on reduction with 10%H_2/He.

Sample Type	Temperature Reduced (° C)	% Concentration			
		V^{5+}	V^{4+}	Zr^{4+}	Al^{3+}
Time	500	8.554	1.006	33.75	56.69
Time	600	7.593	1.457	34.08	56.87
Time	700	7.939	1.541	33.25	57.27
Time	750	7.298	2.162	34.24	56.30
Feed	500	2.950	0.290	7.62	89.14
Feed	600	2.832	0.818	8.61	87.74
Feed	700	2.450	1.010	7.85	88.69
Feed	750	1.161	0.889	7.73	90.22
Conc.	500	0.705	0.075	0.59	98.63
Conc.	600	0.318	0.082	0.58	99.02
Conc.	700	0.314	0.086	0.45	99.15
Conc.	750	0.386	0.464	0.60	98.55