

Memo 2

DIVERSIFIED CHEMICAL PRODUCTS
Research and Product Development Division
Blacksburg VA

TO: U. R. Engineer
FROM: Phil D. Chemist
DATE: 2006 Sep 11
SUBJECT: Results of Kinetic Experiments for Reaction 1

Per your request, we performed a number of batch kinetics experiments in order to provide you with data to evaluate models for the kinetics of the first reaction in the production of the Lucretex monomers.

The experiments were carried out according to the following protocol:

- Charge the 250 mL flask with 41.7 mL of toluene.
- Charge the flask with 35 g of TMDS (R2).
- Place the flask in a constant temperature bath for 1 hour.
- Add 0.22 mL of platinum catalyst slurry (5 mol Pt per L toluene) at $t = 0$.
- Add X g of allyl alcohol (R1) at $t = 0$.
- Take 1 mL samples from the flask every 24 min. These samples are immediately quenched in ice water. Concentration analyses are done later to determine the concentrations of R1, R2, A, and C.
- The reaction is allowed to run for 4 hours.

We were unable to accurately measure the hydrogen evolution rate as you requested. Everything had to be done with extreme care because the materials we were dealing with are toxic. In addition, the temperatures that were requested were above the boiling points of the initial reaction mixtures. Therefore, trials were done at lower temperatures.

The following runs were done: three sets of experiments were done, each set at a particular temperature, leading to a total of nine runs. The temperatures investigated were 80, 85 and 90°C. In each set, the amount of R1 charged into the flask, X, was varied. X took values of 15.135, 30.27, and 45.405. These values correspond to molar ratios of 1:1, 2:1, and 3:1 (R1:R2). Two final experiments were conducted at 85°C with X = 30.27 g, with catalyst loadings of 0.11 and 0.33 mL.

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REACTION I KINETICS STUDY

Catalyst Loading: Base Amount: 0.22 mL (Concentrations in millimolar)

Time, h	R1/R2=1/1				R1/R2=2/1				R1/R2=3/1			
	R1	R2	A	C	R1	R2	A	C	R1	R2	A	C
Temp=80°C												
0.0	2458	2457	0	0	4211	2105	0	0	5526	1842	0	0
0.4	1601	1759	158	26	2984	1208	330	29	4167	912	429	28
0.8	1088	1437	349	59	2261	837	681	60	3400	556	840	54
1.2	781	1264	483	85	1838	646	913	82	2977	382	1089	72
1.6	589	1162	573	107	1573	533	1066	101	2726	284	1242	86
2.0	463	1097	634	128	1395	460	1170	118	2568	223	1340	98
2.4	377	1053	677	149	1270	409	1245	134	2461	183	1406	108
2.8	315	1023	708	169	1178	373	1301	149	2387	155	1452	117
3.2	270	1001	732	189	1109	346	1343	164	2334	135	1485	125
3.6	236	986	750	209	1055	326	1377	177	2294	120	1510	132
4.0	211	974	764	228	1013	311	1404	189	2264	109	1529	138
Temp=85°C												
0.0	2458	2457	0	0	4211	2105	0	0	5526	1842	0	0
0.4	1349	1572	224	50	2635	989	460	55	3793	699	590	51
0.8	815	1244	430	105	1892	626	840	103	3029	366	1022	91
1.2	541	1089	548	149	1511	458	1053	140	2669	226	1241	119
1.6	386	1003	617	189	1289	364	1181	172	2476	153	1362	140
2.0	291	950	660	227	1149	306	1265	202	2362	111	1434	158
2.4	229	916	687	265	1049	266	1323	228	2290	85	1480	172
2.8	187	893	706	301	979	238	1365	252	2242	68	1511	184
3.2	158	877	720	336	926	218	1397	273	2208	56	1532	193
3.6	136	865	730	369	886	202	1422	292	2185	48	1548	200
4.0	120	857	737	400	855	191	1442	308	2167	42	1559	205
Temp=90°C												
0.0	2458	2457	0	0	4211	2105	0	0	5526	1842	0	0
0.4	1086	1378	293	92	2278	771	599	97	3422	497	759	89
0.8	580	1069	489	181	1579	442	969	173	2734	216	1166	148
1.2	357	937	580	255	1263	305	1148	233	2460	115	1339	188
1.6	243	868	626	325	1089	231	1248	285	2328	68	1424	218
2.0	177	828	651	390	981	186	1311	328	2257	44	1471	239
2.4	136	802	667	450	909	157	1354	364	2215	31	1499	253
2.8	109	785	677	505	858	137	1385	395	2190	22	1517	263
3.2	90	773	684	554	820	122	1408	419	2173	17	1528	270
3.6	77	765	688	599	792	111	1425	440	2162	14	1536	275
4.0	67	759	692	639	770	103	1439	457	2155	11	1540	278

Catalyst Loading: Low: 0.11 mL

R1/R2 = 2/1

Temperature = 85°C

Catalyst Loading: High: 0.33 mL

R1/R2 = 2/1

Temperature = 85°C

Time, h	R1	R2	A	C
0.0	4215	2107	0	0
0.4	2948	1215	376	44
0.8	2234	857	731	87
1.2	1836	674	946	120
1.6	1593	566	1081	148
2.0	1433	496	1171	172
2.4	1321	448	1234	195
2.8	1240	413	1281	215
3.2	1179	387	1316	232
3.6	1132	368	1344	248
4.0	1095	353	1365	261

Time, h	R1	R2	A	C
0.0	4208	2104	0	0
0.4	2493	885	496	60
0.8	1753	529	881	110
1.2	1386	372	1090	149
1.6	1176	286	1214	183
2.0	1043	233	1295	214
2.4	952	198	1350	243
2.8	887	173	1390	268
3.2	839	155	1421	291
3.6	801	142	1445	311
4.0	772	132	1464	328

COMPARISON OF CONVERSIONS AND YIELDS AFTER 4.0 HOURS IN THE REACTOR

R1/R2	TEMP, °C	CAT, mL	MOLS A/R2	MOLS C/R2	TOTAL YIELD	MOLS A/C
1:1	80.0	0.22	0.3116	0.0927	40.43%	3.361
	85.0	0.22	0.3009	0.1633	46.42%	1.843
	90.0	0.22	0.2825	0.2600	54.25%	1.087
2:1	80.0	0.22	0.6678	0.0907	75.85%	7.363
	85.0	0.22	0.6862	0.1472	83.34%	4.662
	90.0	0.22	0.6847	0.2177	90.24%	3.145
3:1	80.0	0.22	0.8321	0.0746	90.67%	11.154
	85.0	0.22	0.8490	0.1109	95.99%	7.656
	90.0	0.22	0.8382	0.1512	98.94%	5.544
2:1	85.0	0.11	0.6494	0.1230	77.24%	5.280
2:1	85.0	0.33	0.6958	0.1559	85.22%	4.463