

## APPENDIX A

### EXPERIMENTAL DATA

The data presented in Appendix A represents the majority of the successful runs performed in support of this project. Data has been omitted where runs were either too short to be meaningful (due to equipment malfunctions) or the data was incomplete for the format used here (this was the case with the earliest runs). This data has been selected for inclusion because it is the basis for essentially all of our conclusions and recommendations. While the layout of the data on each page is fairly self-explanatory, a few clarifying comments are warranted:

1. Due to our instrumentation, no single consistent set of units has been used. Thus, pressures are in psia, temperatures in degrees centigrade, gas flows in liters per minute, and liquid volumes in milliliters. The average flow rates were determined over 10 minute intervals due to fluctuations in the flow which affected the reliability of more frequent measurements.
2. Except for values which were read directly from instruments (system pressure, temperature, cumulative outlet of both water and gas, and gas compositions) the number of significant figures included does not reflect the precision of the experiment. The extra significant figures were included for calculational purposes only, and are an artifact of the data processing methods employed.
3. The "water in" and "water out" readings are cumulative.
4. The normalized gas composition data has been adjusted to exclude non-reacting species (oxygen and nitrogen). The nitrogen is residual material from startup of the system and the oxygen is attributed to sample handling by syringe.
5. The water consumption has been estimated based upon the reaction stoichiometry and the calculated average reaction rate. The liquid volume in the reactor has been estimated based upon the density of pure water under the reaction temperature. Agreement between estimated and measured ending liquid volumes was generally within 10%.
6. The units for the calculated values are shown in most cases. Where these units are not indicated alongside the data, they are as follows: mol/liter·hr psi for average and standard deviation of the k values on the row above.

7. The following catalysts and conditions were used in the experiments:

<u>Run #</u>	<u>Catalyst</u>	<u>temp (°C)</u>	<u>press psig</u>
7	0.6M potassium carbonate	250	1000
8	0.6M potassium carbonate	250	1000
15	0.6M sodium carbonate	320	2500
16	0.6M sodium carbonate	330	2500
17	0.6M sodium carbonate	337	2500
18	0.6M sodium carbonate	300	2400
19	0.6M sodium carbonate	300	2500
20	0.6M sodium carbonate	300	2000
22	0.6M sodium carbonate	300	3000
23	0.6M sodium carbonate	330	3000
24	0.6M sodium carbonate	350	3000
25	0.6M sodium citrate	300	3000
26	0.6M cadmium hydroxide	300	3000
27	no catalyst	300	3000
28	no catalyst	300	3000
29	0.6M sodium carbonate	300	3000
30	0.6M ammonium hydroxide	300	3000
31	0.6M ammonium hydroxide	300	3000

The intent of this appendix is to give the reader adequate information to be able to check any of the results or conclusions presented in the body of the report. To this end, every attempt was made to present raw data, rather than pre-processed information.

run #	catalyst temperature	7	250	dip tube experiments
initial catalyst vol. (cc)	470			
pressure	2550			standard dip tube
water density @ temp.	0.799938			
water vap. press. @ temp.	5.48			
system pressure				
time	1000	1008	1005	1006
outlet flow	0.1	20	60	100
avg outlet flow (liter/min)	0.38	1.5	1.47	1.76
cumulative outlet	0.1	1.205527	1.5	0.78
H2	0.01	24	54	70.72
C02	0	0.67	1.62	2.6
C0	0	25.69	3.11	2.82
water out	1	61.08	92.29	91.25
water in	1	1	1	1
10 min avg inlet	0	0	0	0
fractional conv.	0.1	1.025816	1.463455	0.812563
liq. vol.	0	0.177484	0.024985	0.028842
10 min avg rate	586.7648	585.8390	583.4899	582.9184
C0 overpressure	0	0.771001	0.155768	0.099937
turnover	4.52	280.968	424.534	417.0125
k	0	81.96895	16.49e07	10.57184
	0.0003146545	0.000366	0.000239	0.000270
normalized H2	0.0007662	0.016697	0.026895	0.031333
" C02	0.0293801	0.03055	0.029171	0.032158
" C0	1.0698536	0.951247	0.943932	0.936507
avg. of C02 & H2	0	0.150731	0.023376	0.028033
water consumed (grams)	0	0.39981	2.617665	3.074487
10 min avg molar inlet	0.0004142	0.042415	0.060627	0.033663
	0.000694861			

run #	8	catalyst temperature	250	dip tube experiments
initial catalyst vol. (cc)	470	4" x.0625" dip tube	994	991
pressure	2550	4" x.0625" dip tube	170	190
water density @ temp.	0.779298			
water vap. press. @ temp.	548			
system pressure	981	991	984	986
time	0.1	20	60	80
outlet flow	1.24	0.52	0.66	0.57
avg outlet flow (liter/min)	0.1	0.681407	0.26625	0.5845
cumulative outlet	0.01	13.57	24.22	35.91
H2	0	0.34	1.82	3.12
C02	0	34.73	18.41	9.86
C0	1	18.62	62.22	78.51
water out	0	0	0	0
water in	0	0	0	0
10 min avg inlet	0.1	0.458861	0.233586	0.543037
frictional corr.	0	0.484995	0.139835	0.076352
liq. vol.	588.0159	586.7579	582.7213	581.9381
10 min avg rate	0	0.942781	0.139333	0.117704
C0 overpressure	4.33	82.4866	273.1458	342.2036
turnover	0	100.3289	14.73437	18.70348
k	0.011429	0.000510	0.000517	0.000353
	0.00003091377			
normalized H2	0	0.006332	0.022073	0.034102
" CO2	0	0.646861	0.222886	0.10771
" C0	1	0.346805	0.754639	0.858126
aug. of CO2 & H2	0	0.326597	0.122680	0.070936
water consumed (grams)	1.005557	4.231981	4.857997	5.398564
10 min avg molar inlet	0.004142	0.019009	0.009677	0.022497

run #	15
catalyst temperature	320
initial catalyst vol. (cc)	470
pressure	2550
water density @ temp.	0.670292
water vap. press. @ temp.	1602
system pressure (psig)	2548
time (min)	2553
outlet flow (liter/min)	10
avg. outlet (liter/min)	1.94
cumulative outlet	2.54
H2	25.4
C02	0.314
CO	0.532
water out (ml)	2.017
water in (ml)	13
	0
inlet flow (liter/min)	1.653370 4.313967 3.500772 2.631288 2.764734 4.895995 4.748256 2.516686 2.732833 0.840159 0.252484 0.144981
10 min avg inlet	2.164722 3.181550 3.547734 3.299388 2.784482 3.803139 5.236272 2.880208 2.743185 1.142966 0.385039 0.181226
fractional conv.	0.173360 0.159025 0.171170 0.216134 0.265944 0.143791 0.132559 0.192043 0.207537 0.428300 0.584233 0.655387
liq. vol.	681.7915 654.0039 649.1034 657.7463 674.7289 620.9254 491.9182 451.7142 465.0852 522.4426 573.5116 571.5592
aug rate	1.368207 1.922991 2.325497 2.711433 2.722079 2.189214 3.631803 3.043596 3.055923 2.329134 0.975023 0.516546
CO overpressure (psia)	19.08082 643.7319 663.5739 604.6785 545.5878 698.2631 708.9242 635.3496 613.2772 371.3187 242.0145 193.2368
turnover (mol/liter hr)	79.56417 107.2683 128.7489 151.1896 157.0002 115.9422 152.3804 117.2703 120.7028 103.7879 47.69481 25.18166
k (mol/hr liter psi)	0.071705 0.002987 0.003504 0.004484 0.004999 0.003135 0.005122 0.004190 0.004982 0.006272 0.004028 0.002673
normalized H2	0.109675 0.122199 0.139648 0.173405 0.204354 0.126685 0.123530 0.164340 0.166903 0.314720 0.390306 0.434557
" CO2	0.185819 0.152213 0.152657 0.182039 0.215797 0.124744 0.117855 0.157868 0.176334 0.285014 0.342269 0.357266
" CO	0.704505 0.725586 0.707593 0.6449555 0.579848 0.749369 0.758613 0.677791 0.655626 0.400265 0.262924 0.208176
avg. of CO2 & H2	0.147747 0.157206 0.14653 0.177722 0.210075 0.125715 0.120693 0.161104 0.171868 0.299867 0.368787 0.395911
water consumed (grams)	6.625777 14.91059 24.79846 35.73400 45.79811 55.27069 70.21915 78.59726 87.81042 93.57922 94.88788
avg Molar inlet	0.089681 0.131807 0.146977 0.136688 0.115357 0.157558 0.216931 0.119322 0.113646 0.047351 0.015951 0.007307
0.0010541029	

run #	16
catalyst temperature	330
initial catalyst vol. (cc)	470
pressure	2540
water density @ temp.	0.640661
water vap. press. @ temp.	1836
system pressure	2541
time	10      30      50      80      120      140      160      210      230      2540      2541      2542      2543      2544      2545      2546      2547      2548      2549      2540      2538      2537
outlet flow	2.9      1.2      0.13      2.6      2.66      2.8      1.34      1.43      1.8      1      1.05      1.07
avg. outlet	3.16      1.949      0.896      1.754      2.6815      2.5625      2.0975      1.369      1.5235      1.303333      0.955      1.06
cumulative flow	31.6      70.58      88.5      141.12      194.75      246      287.95      315.33      345.8      384.9      404      414.6
H2	1      22.1      36.97      26.43      30.98      30.37      36.32      39.65      39.06      43.75      44.48      43.41
CO2	1.455      31.61      37.58      31.96      30.99      31.23      32.42      37.27      39.73      38.61      40.13      41.06
CO	8.204      33.2      16.96      39.56      36.21      36.49      29.17      21.11      19.22      16.57      14.36      14.17
water out	10      10      10      24      30      30      30      30      30      30      30      30      30
water in	0      0      0      0      50      50      65      65      95      95      95      95
inlet flow	2.566033      0.829202      0.077046      1.825043      1.820520      1.920807      0.869610      0.869001      0.855213      1.050742      0.572547      0.600418
10 Min avg inlet flow	2.796091      1.346763      0.531050      1.231202      1.835235      1.757881      1.361200      0.831931      0.911131      0.760815      0.546782      0.606136
fractional conc.	0.130148      0.447173      0.687286      0.424623      0.461120      0.457720      0.540919      0.645567      0.672097      0.713073      0.746580      0.748781
Liq. vol.	718.2326      704.3085      691.9066      652.9364      705.8248      686.6044      668.4708      676.7427      663.3715      669.9888      678.8773      673.8589
10 Min avg rate	1.259441      2.125470      1.311174      1.990277      2.980301      2.912957      2.737933      1.972680      2.294601      1.954441      1.494689      1.674199
CO overpressure	57.8382      234.724      118.5504      278.898      256.3668      257.9843      206.2319      149.0366      135.501      116.6528      100.8072      99.3337
turnover (mol/liter hr)	77.15373      127.6827      77.37878      110.8404      179.4200      170.5904      156.1058      113.8660      129.8308      115.0214      86.54785      96.22558
k (mol/liter hr psi)	0.021775      0.009055      0.011060      0.007136      0.011625      0.011291      0.013275      0.013236      0.016934      0.016754      0.014827      0.016654
0.0129136759	
0.0031092073	
normalized H2	0.093817      0.254286      0.403999      0.269831      0.315542      0.309613      0.370952      0.404346      0.398530      0.449429      0.440085
" CO2	0.136504      0.363709      0.410665      0.326288      0.315644      0.318381      0.331120      0.380267      0.405346      0.405476      0.416261
" CO	0.769678      0.382094      0.185334      0.403879      0.368812      0.372005      0.297926      0.215386      0.196102      0.167492      0.145094      0.143653
avg. of CO2 & H2	0.115160      0.308997      0.407332      0.298060      0.315953      0.313997      0.351036      0.392306      0.401948      0.416253      0.427552      0.428173
water consumed (grams)	8.917852      16.86075      27.81964      37.94672      50.25660      61.87049      71.57269      80.13636      93.08903      100.2055      103.4196
10 min avg Molar inlet	0.115837      0.055794      0.021999      0.051006      0.076031      0.072826      0.056392      0.034465      0.037746      0.031519      0.022652      0.025111

run #	17	stir rate=2
catalyst temperature	337	
initial catalyst vol. (cc)	470	
pressure	2540	
water density @ temp.	0.619283	
water vap. press. @ temp.	2015.9	
system pressure	2533	2537
time	10	30
outlet flow	1.9	2.95
avg flow (liter/min)	1.61	2.195
cumulative flow	16.1	60
H2	0.26	23.05
C02	0.877	33.93
C0	0	28.95
water out	0	0
water in	0	0
inlet flow	0.95	1.971930
10 min avg inlet flow	0.805	1.467250
fractional conv.	1	0.495995
liq. vol.	758	9419
10 min avg rate	2.636563	2.449609
C0 overpressure	0	150.8584
turnover (liter/mole hr)	169.4736	153.2105
k (mol/liter hr psi)	ERR 0.016237	0.0117346
	0.0241301938	0.0095652761
normalized H2	0.228671	0.268241
" C02	0.771328	0.394856
" CO	0	0.336902
avg. of C02 & H2	0.5	0.331548
water consumed (grams)	12.67443	25.49303
10 min avg molar flow	0.033349	0.060786

run #	19
catalyst temperature	300
initial catalyst vol. (cc)	470
pressure	2600
water density @ temp.	0.71222
water vap. press. @ temp.	1205
system pressure	2573
time	20
outlet flow	3.47
avg. flow (liter/min)	2.9625
cumulative outlet	59.25
H2	4.202
CO2	5.279
CO	46.52
Water out	47
water in	40
10 min avg inlet	2.711723
fractional conv.	0.092478
liq. vol.	650.0800
10 min avg rate	0.958894
CO overpressure	636.3936
turnover (liter/mole hr)	53.16814
k (mol/liter hr psi)	0.001506
	0.0014502617
normalized H2	0.075034
" CO2	0.094266
" CO	0.830699
" H2	0.084650
avg. of CO2 & H2	4.713424
water consumed (grams)	0.112342
10 min avg inlet molar	0.128937
	0.0001651111

run #	20
catalyst temperature	300
initial catalyst vol. (cc)	470
pressure	2040
water density @ temp.	0.71222
water vap. press. @ temp.	1205
System pressure	2021
time	2016
outlet flow	40
avg outlet flow (liter/min)	2.3325
cumulative outlet	2.9525
H2	105.7
C02	5.171
C0	5.992
water out	81.64
water in	65
	50
10 min avg inlet	2.163606
fractional conv.	0.073061
liq. vol.	638.3475
10 min avg rate	0.657152
C0 overpressure	666.4824
turnover (liter/mol hr)	35.80778
k (mol/liter psi hr)	0.000986
	0.0001711947
	0.000415333
10 min avg inlet	2.784073
2.682999	1.802157
2.111874	2.012564
2.082963	1.253930
1.174723	0.776884
0.738100	0.742259
0.497640	0.344295
0.341330	0.458048
0.224117	0.293628
0.166606	0.166606
0.157727	0.157727
0.145901	0.145901
0.105619	0.105619
0.061312	0.061312
0.060496	0.060496
0.061240	0.061240
0.054148	0.054148
0.045545	0.045545
" CO2	0.09271
" CO	0.851182
avg. of CO2 & H2	0.072408
water consumed (grams)	2.556077
10 min avg molar inlet	0.089635

normalized H2	0.045545
CO2	0.09271
" CO	0.851182
avg. of CO2 & H2	0.072408
water consumed (grams)	2.556077
10 min avg molar inlet	0.089635



run #	23
catalyst temperature	330
initial catalyst vol. (cc)	470
pressure	2950
water density @ temp.	0.640461
water vap. press. @ temp.	1836
system pressure	2966
time	20
outlet flow	40
avg outlet flow (liter/min)	4.55
cumulative flow	3.4625
H2	69.25
C02	12.92
C0	22.33
water out	40.21
water in	65
inlet flow by 10 min avg	50
fractional conv.	191728
liq. vol.	3.165374
rate (10 min avg)	2.979114
C0 overpressure	2.849777
turnover (Mol/litter hr)	1.359193
k (Mol/litter hr psi)	1.288351
0.006227	1.159764
0.006852	1.086883
0.006202	0.647783
0.006547	0.534638
0.006229	0.462487
0.006852	0.590484
0.006202	0.546532
0.006546	0.555530
normalized H2	0.171216
" C02	0.29518
" CO	0.532865
avg. of C02 & H2	0.233567
water consumed (grams)	13.69795
Molar inlet (10 min avg)	0.109941
0.0032297676	0.0177737
normalized H2	0.231570
" C02	0.257552
" CO	0.510877
avg. of C02 & H2	0.244561
water consumed (grams)	13.69795
Molar inlet (10 min avg)	0.132228
0.01136	0.011131
0.010196	0.010131
0.007279	0.007279
0.006547	0.006547
0.006202	0.006202
0.006852	0.006852
0.009505646	0.009505646

run #	24
catalyst temperature	350
initial catalyst vol. (cc)	470
pressure	2950
water density @ temp.	0.5743
water vap. press. @ temp.	2380
system pressure	2995
time	20
outlet flow	2.7
avg outlet flow (liters/min)	2.365
cumulative flow	47.3
H2	18.6
CO2	18.9
CO	7.396
water out	48
water in	68
inlet flow by 10 min avg	1.377300
fractional conv.	0.717126
liq. vol.	85.2126
rate (10 min avg)	2.877520
CO overpressure	45.4854
turnover (mole/liter hr)	209.4062
k (mol/liter hr ps)	0.063262
0.039027511	0.0132386337
normalized H2	0.414290
" C02	0.420972
" CO	0.484736
avg. of C02 & H2	0.417631
water consumed (grams)	18.28512
molar inlet (10 min avg)	0.057059

run #	25	Sodium Citrate
catalyst temperature	300	
initial catalyst vol. (cc)	470	
pressure	2950	
water density @ temp.	0.712224	
water vap. press. @ temp.	1205	
system pressure	3003	2979
time	20	40
outlet flow	3.3	3.16
avg outlet flow (liter/min)	3.1275	2.9625
cumulative flow	62.55	121.8
H2	1.394	11.32
CO2	15.11	14.04
CO	40.46	63.13
water out	28	53
water in	25	55
inlet flow by 10 min avg	2.674439	2.537794
fractional cond.	0.169403	0.167260
liq. vol.	655.6925	653.5260
rate (10 min avg)	1.717539	1.614623
CO overpressure	727.4708	1119.926
turnover (mol/liter hr)	96.055519	90.001118
k (mol/liter hr psi)	0.002360	0.001441
0.002115997	0.0005574508	0.002482
normalized H2	0.024471	0.127924
" CO2	0.26555	0.158661
" CO	0.710273	0.713413
avg. of CO2 & H2	0.144863	0.143293
water consumed (grams)	6.543039	15.10684
molar inlet (10 min avg)	0.110798	0.105145

run #	26	Cadmium Hydroxide
catalyst temperature	300	
initial catalyst vol. (cc)	470	
pressure	2950	
water density @ temp.	0.712224	
water vap. press. @ temp.	1205	
system pressure	2995	
time	20	
outlet flow	2.63	
avg outlet flow (liter/min)	2.865	
cumulative flow	125.7	
H2	2.83	
CO2	0.682	
CO	32.27	
water out	0	
water in	0	
inlet flow by 10 min avg	2.724400	3.171087
fractional conv.	0.051607	0.078494
liq. vol.	659.9047	655.8893
rate (10 min avg)	0.529607	0.943338
CO turnover (mole/liter hr)	577.633	1432.289
k (mol/liter hr psi)	29.80913	52.77303
	0.000916	0.000658
	0.0005881935	0.0000879287
normalized H2	0.079090	0.074620
" CO2	0.019059	0.070943
" CO	0.901850	0.854436
avg. of CO2 & H2	0.049074	0.072781
water consumed (grams)	2.859868	6.751750
molar inlet (10 min avg)	0.112867	0.131373

run #	catalyst temperature	28	no catalyst	
initial catalyst vol. (cc)	300			
pressure	470			
water density @ temp.	0.712224			
water vap. press. @ temp.	1205			
system pressure	2950			
time	3001	3001	3010	3010
outlet flow	20	40	85	125
outlet flow	2.61	2.8	2.73	2.05
avg outlet flow	3.1105	2.562	2.756	2.365
cumulative flow	62.21	113.45	168.57	238.57
H2	0.638	1.663	2.332	2.671
CO2	0.51	1.678	2.702	3.168
CO	47.07	83.74	90.35	92.34
water out	45	68	95	130
water in	55	75	95	130
inlet flow by 10 min avg	3.074101	2.512852	2.683274	2.716737
fractional conv.	0.011840	0.019558	0.027103	0.030647
liq. vol.	673.9452	668.8147	657.7170	655.6763
rate (10 min avg)	0.134247	0.182661	0.274851	0.313652
CO overpressure	845.3772	1508.994	1622.686	1666.737
turnover (liter/hole hr)	NA	NA	NA	NA
k (mol/liter hr psi)	0.000158	0.000121	0.000169	0.000189
0.0001811623	0.0000279326	0.0000196	0.0000227	0.0000183
normalized H2	0.012822	0.019097	0.024448	0.027205
" CO2	0.010581	0.019269	0.028327	0.032267
" CO	0.976596	0.961653	0.947223	0.940526
avg. of CO2 & H2	0.011701	0.019183	0.026388	0.027736
water consumed (grams)	0.654078	1.550108	3.011583	4.237218
molar inlet (10 min avg)	0.127355	0.104103	0.111164	0.112550

run #		30			
catalyst temperature		300			
initial catalyst vol. (cc)		470			
pressure		2600			
water density @ temp.	0.71222				
water vap. press. @ temp.	1205				
system pressure	3011				
time	20	40	75	108	
outlet flow	3	3			
avg outlet flow (liter/min)	2.905	2.77	0.68	3.184848	
cumulative outlet	58.1	113.5	137.3	242.4	
H2	8.035	22.68	25.99	23.29	
CO2	1.133	21	23.85	22.69	
CO	34.39	46.33	37.78	50.7	
water out	26	45	45	49	
water in	25	45	45	45	
10 min avg inlet	2.867218	2.446869	0.587452	2.811119	
fractional conv.	0.200074	0.417305	0.500891	0.405370	
liq. vol.	658.5043	642.7329	617.2080	584.9721	
10 min avg rate	2.165432	3.948987	1.183048	4.848227	
CO overpressure	621.0854	832.0868	676.6398	925.275	
turnover (liter/mol hr)	121.6234	216.4861	62.3519	241.8979	
k	0.003486	0.004745	0.001751	0.005239	
normalized H2	0.184466	0.251972	0.296621	0.240897	
" CO2	0.026011	0.233307	0.272198	0.234691	
" CO	0.789522	0.514720	0.431180	0.524410	
avg. of CO2 & H2	0.197472	0.368625	0.432720	0.358243	
Water consumed (grams)	12.23273	30.41207	49.37111		
10 Min avg inlet Molar	6.118784	0.101370	0.024337	0.116460	

run #	31
catalyst temperature	300
initial catalyst vol. (cc)	470
pressure	2600
water density @ temp.	0.71222
water vap. press. @ temp.	1205
system pressure	3000
time	20 40
outlet flow	
avg outlet flow (liter/min)	2.57 2.99
cumulative outlet	
H2	51.4 111.2
CO2	5.57 21.49
CO	6.779 18.5
water out	20.64 45.48
water in	0 0
water in	0 0
10 min avg inlet	
fractional conv.	2.305941 2.566406
liq. vol.	0.302690 0.403306
10 min avg rate	659.9084 641.5306
CO overpressure	2.629147 4.166724
turnover (liter/mol hr)	370.4688 816.8208
k	147.9832 227.9954
	0.007036 0.005101
normalized H2	0.168844 0.251433
" CO2	0.205492 0.216450
" CO	0.625663 0.532116
avg. of CO2 & H2	0.271590 0.359658
water consumed (grams)	13.08900
10 min avg inlet molar	0.095531 0.110465