

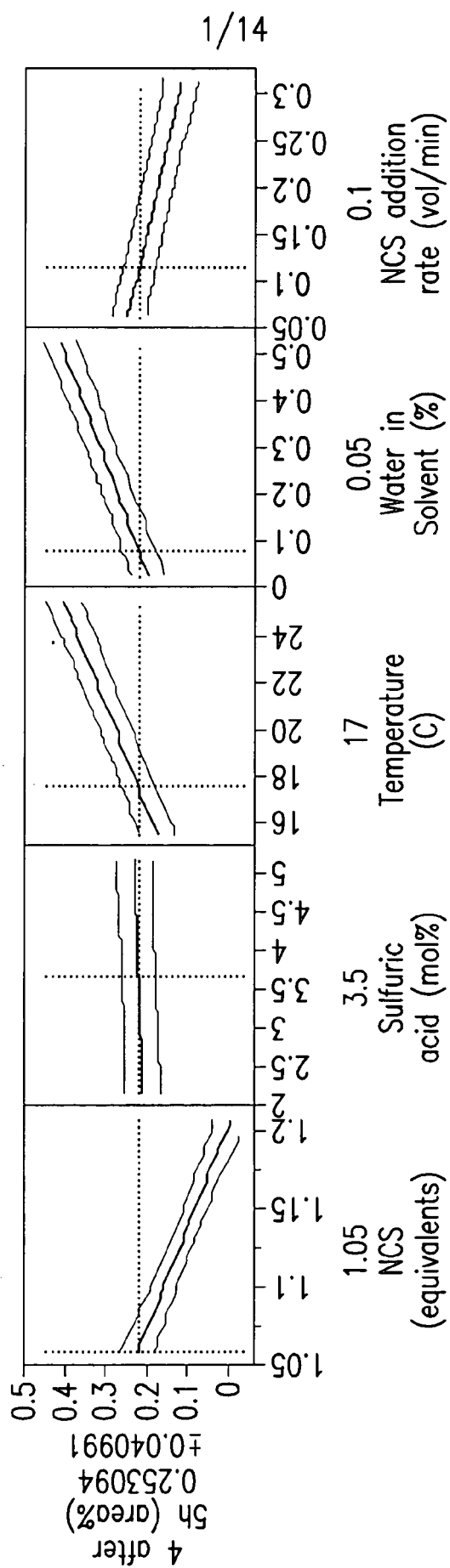


FIG. 1

2/14

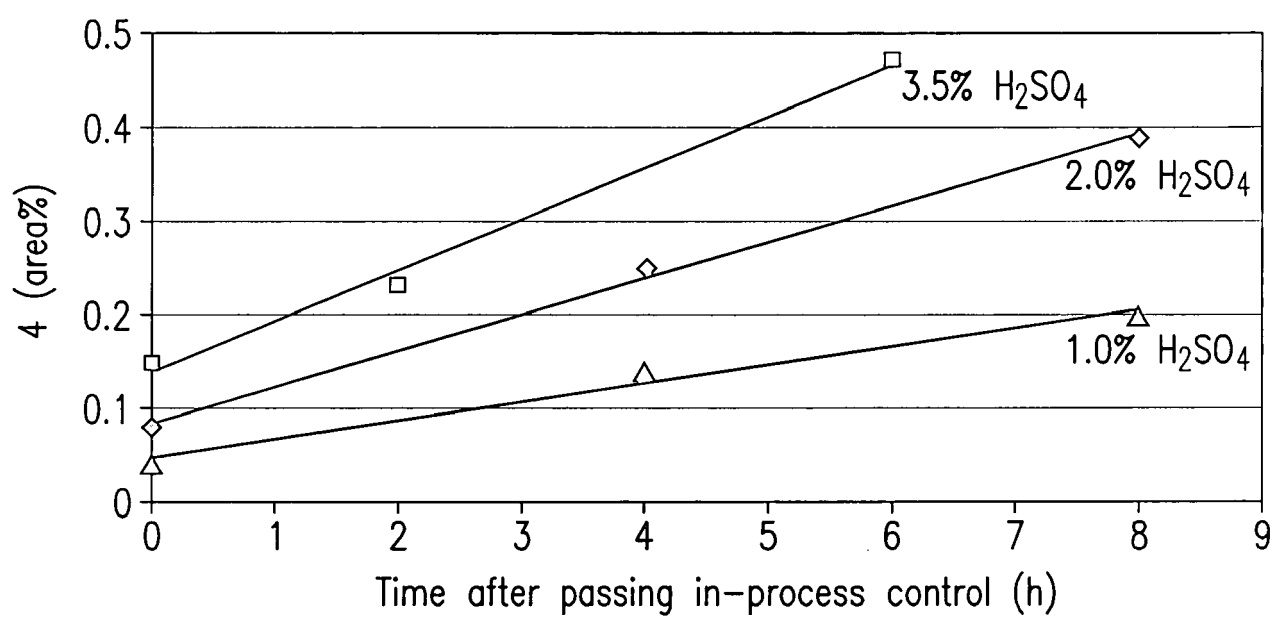


FIG.2

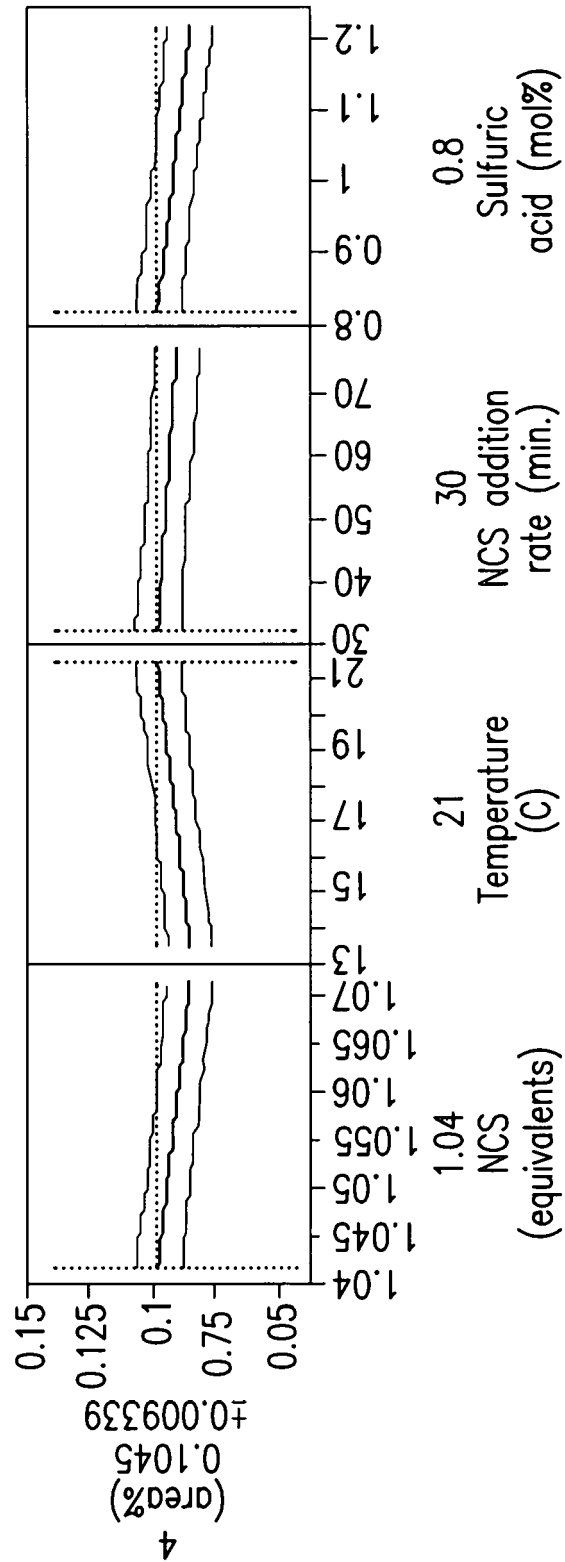


FIG. 3

4/14

Initial Design of Experiment

Experimental Table

Experiment	Pattern	NCS (equivalents)	Sulfuric acid (mol%)	Temp. (C)	Solvent (volume)	Water in solvent (%)	NCS addition rate (vol/min)	Solvent	4 after 5h (area%)
1	--++--+	1.05	2	25	3	0	0.05	MeOAc	0.239
2	++--++-	1.2	5	15	2	0.5	0.3	EtOAc	0.05
3	-++--++	1.05	5	25	2	0	0.3	MeOAc	0.139
4	2	1.125	3.5	20	2.5	0.25	0.175	MeOAc	0.247
5	+--++--	1.2	2	15	3	0.5	0.05	EtOAc	0.068
6	-+-+--	1.05	5	15	3	0	0.3	EtOAc	0.095
7	+++-	1.2	5	25	2	0	0.05	EtOAc	0.125
8	1	1.125	3.5	20	2.5	0.25	0.175	EtOAc	0.192
9	-+--+-	1.05	5	15	2	0.5	0.05	MeOAc	0.234
10	-----	1.05	2	15	2	0	0.05	EtOAc	0.185
11	--+-++-	1.05	2	25	2	0.5	0.3	EtOAc	0.475
12	+--++--	1.2	2	25	3	0	0.3	EtOAc	0.123
13	+--+-++	1.2	2	25	2	0.5	0.05	MeOAc	0.322
14	+--++--	1.2	2	15	2	0	0.3	MeOAc	0.129
15	++-+-++	1.2	5	15	3	0	0.05	MeOAc	0.168
16	---+++-	1.05	2	15	3	0.5	0.3	MeOAc	0.11
17#	1	1.125	3.5	20	2.5	0.25	0.175	EtOAc	0.372
18	++++++	1.2	5	25	3	0.5	0.3	MeOAc	0.472
19	---++--	1.05	5	25	3	0.5	0.05	EtOAc	0.68

#Experiment 17 was deemed an outlier and was ignored for the analysis

FIG. 4

5/14

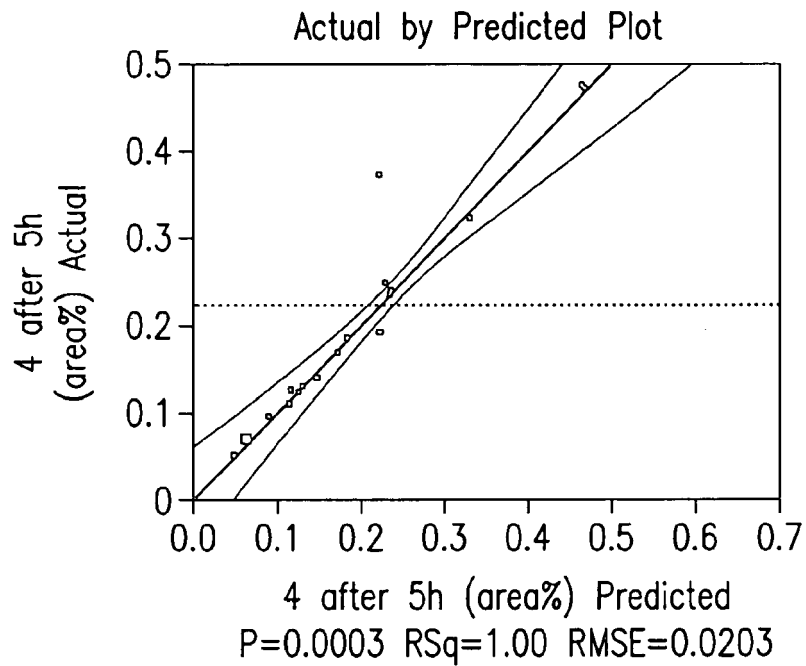


FIG.5A

Summary of Fit	
RSquare	0.9965
RSquare Adj	0.985126
Root Mean Square Error	0.020258
Mean of Response	0.225167
Observations (or Sum Wgts)	18

FIG.5B

Analysis of Variance				
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	13	0.46741489	0.035955	87.6090
Error	4	0.00164161	0.00410	Prob>F
C. Total	17	0.46905650		0.0003*

FIG.5C

6/14

Parameter Estimates				
Term	Estimate	Std Error	t Ratio	Prob> t
Intercept	0.2251667	0.004775	47.16	<.0001*
NCS (equivalents)(1.05,1.2)	-0.04375	0.005065	-8.64	0.0010*
Sulfuric acid (mol%)(2,5)	0.0195	0.005065	3.85	0.0183*
Temperature (C)(15,25)	0.096	0.005065	18.96	<.0001*
Solvent (vol)(2,3)	0.0185	0.005065	3.65	0.0217*
Water in Solvent (%)(0,0.5)	0.0755	0.005065	14.91	0.0001*
NCS addition rate (vol/min)(0.05,0.3)	-0.02675	0.005065	-5.28	0.0062*
Solvent[EtOAc]	-0.003722	0.004775	-0.78	0.4792*
NCS (equivalents)*Temperature (C)	-0.017625	0.005065	-3.48	0.0254*
NCS (equivalents)*Solvent (vol)	0.007125	0.005065	1.41	0.2322*
NCS (equivalents)*Water in Solvent (%)	-0.029625	0.005065	-5.85	0.0043*
NCS (equivalents)*NCS addition rate (vol/min)	0.038125	0.005065	7.53	0.0017*
NCS (equivalents)*Solvent[EtOAc]	-0.089875	0.005065	-17.75	<.0001*
Sulfuric acid (mol%)*Temperature (C)	0.012625	0.005065	2.49	0.0673

FIG.5D

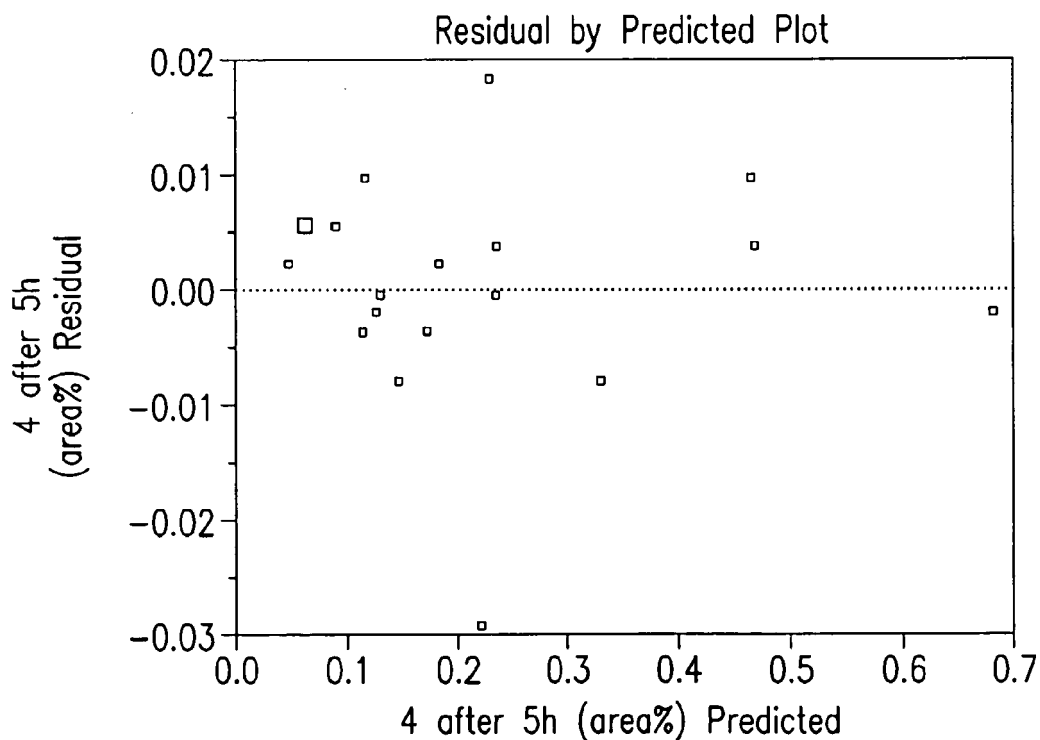


FIG.5E

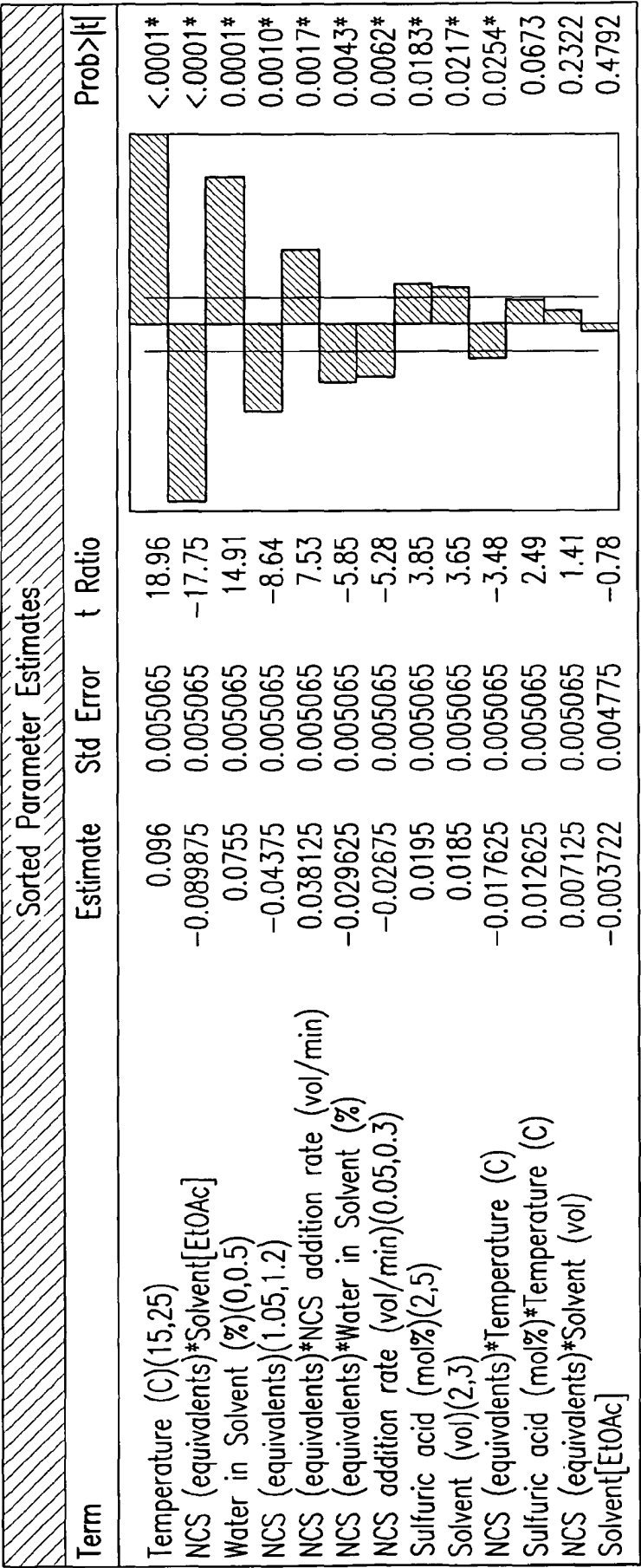


FIG. 5F

Prediction Profiler

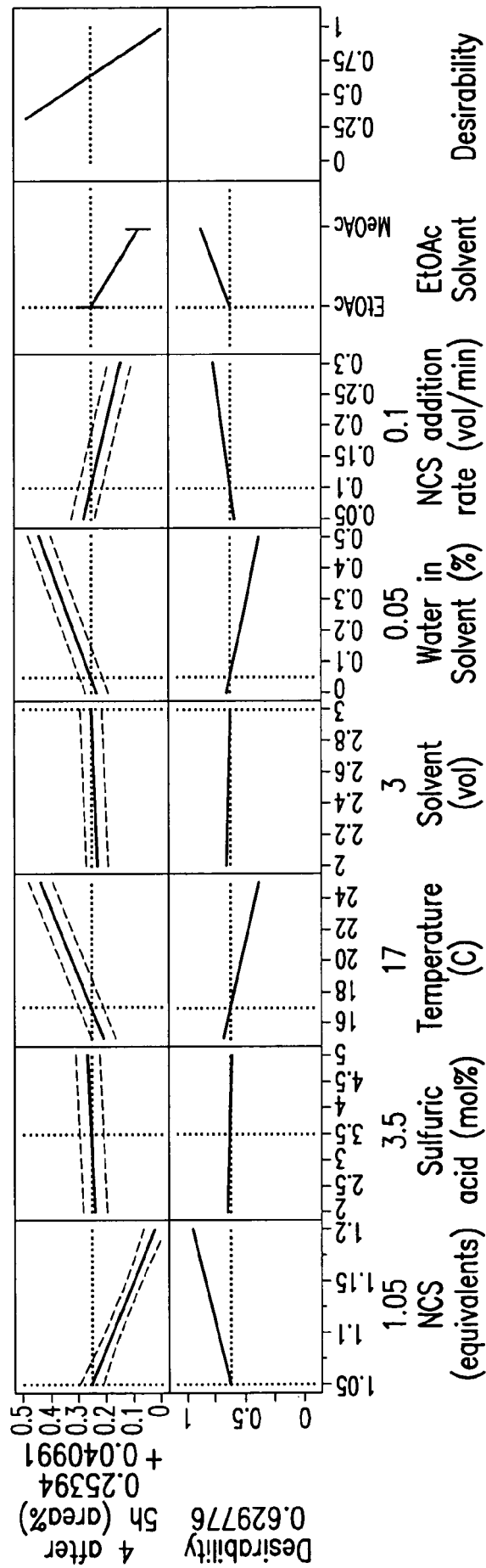


FIG.6A

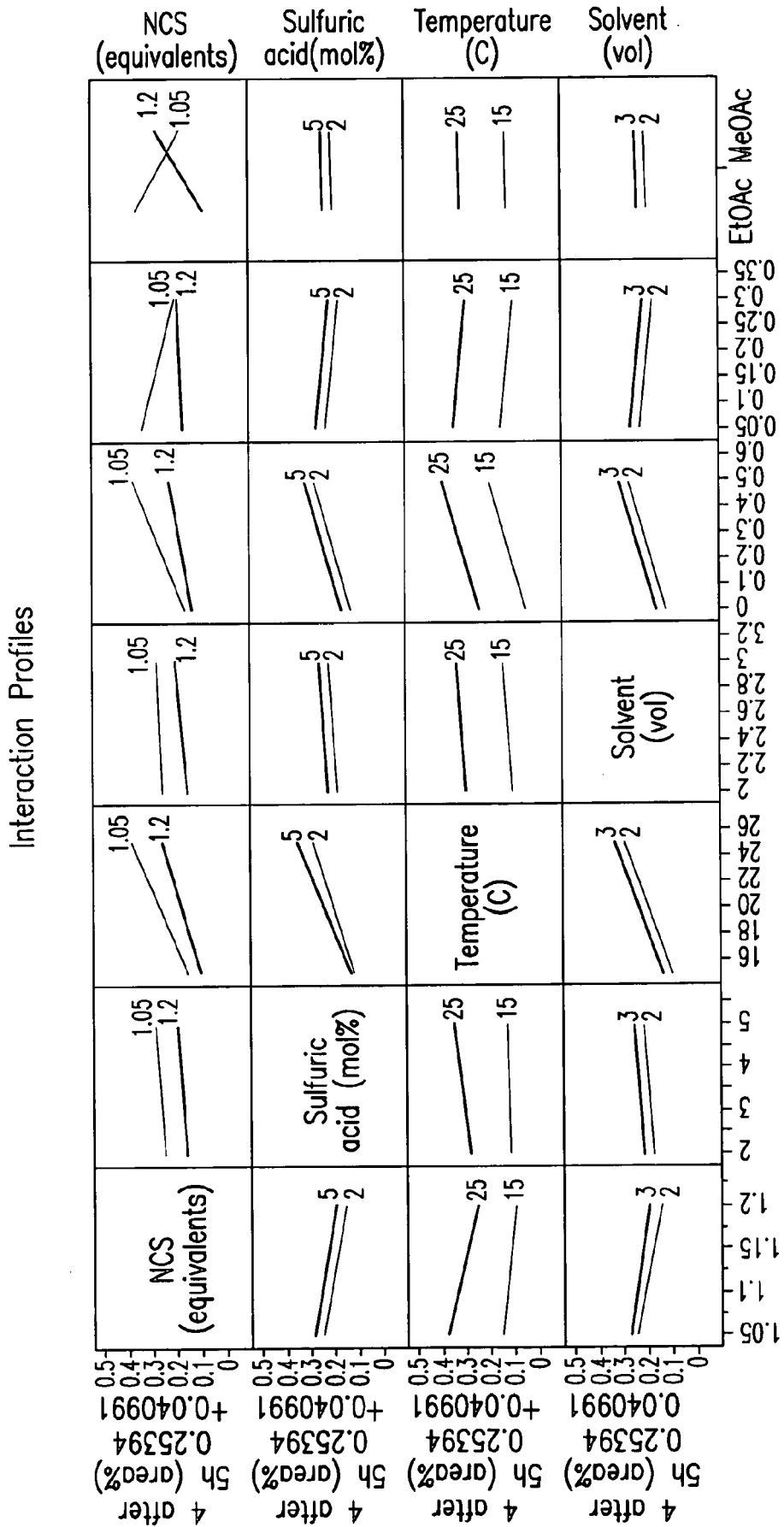


FIG. 6B

Interaction Profiles

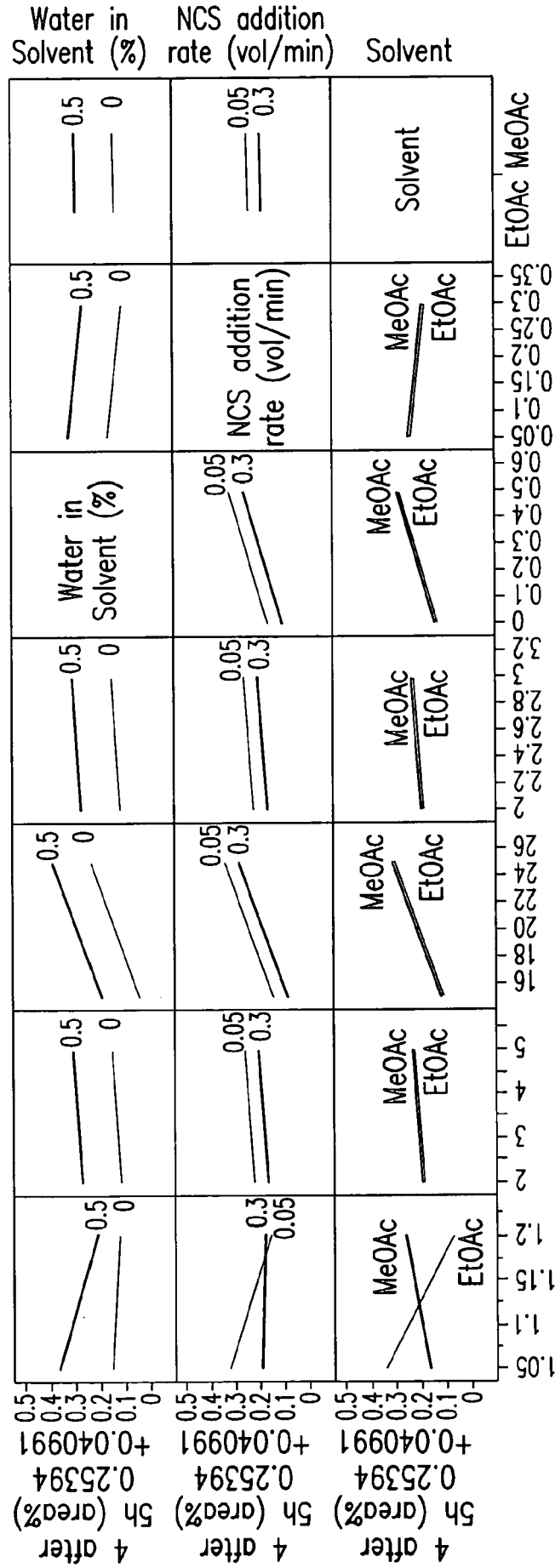


FIG. 6B-1

11/14

Robustness Design of ExperimentExperimental Table

Experiment	Pattern	NCS (equivalents)	Temp. (C)	NCS addition rate (vol/min)	Sulfuric acid (mol%)	4 (area%)
1	+--+	1.07	13	75	0.8	0.07
2	0	1.055	17	52.5	1	0.09
3	----	1.04	13	30	0.8	0.09
4	-+-+	1.04	21	30	1.2	0.09
5	--++	1.04	13	75	1.2	0.07
6	++++	1.07	21	75	1.2	0.07
7	+--+	1.07	13	30	1.2	0.07
8	0	1.055	17	52.5	1	0.08
9	-++-	1.04	21	75	0.8	0.1
10	++--	1.07	21	30	0.8	0.09

FIG.7

12/14

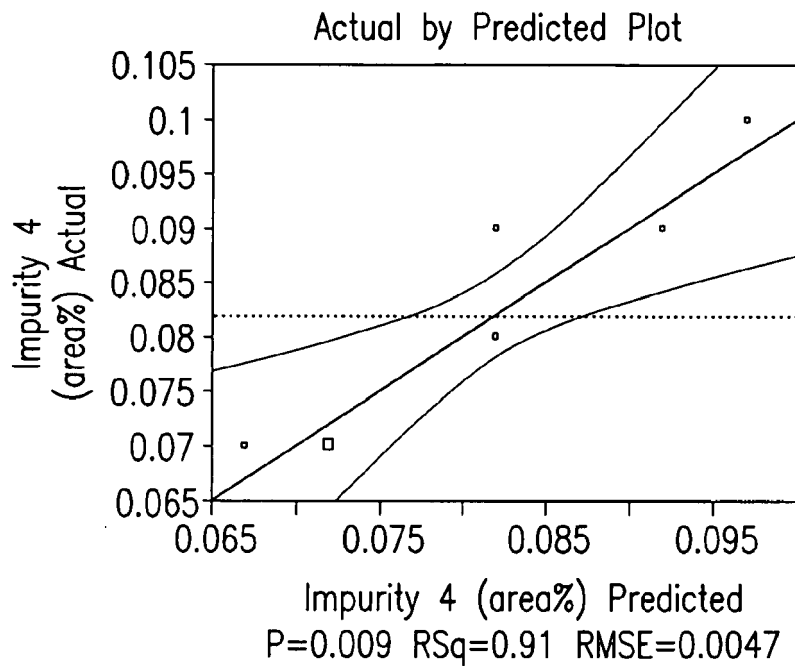


FIG.8A

Summary of Fit	
RSquare	0.905172
RSquare Adj	0.82931
Root Mean Square Error	0.00469
Mean of Response	0.082
Observations (or Sum Wgts)	10

FIG.8B

Analysis of Variance				
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	4	0.00105000	0.000262	11.9318
Error	5	0.00011000	0.000022	Prob>F
C. Total	9	0.00116000		0.0090*

FIG.8C

13/14

Lack Of Fit				
Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	4	0.00006000	0.000015	Prob>F
Pure Error	1	0.00005000	0.000050	0.8581
Total Error	5	0.00011000		Max RSq
				0.9569

FIG.8D

Parameter Estimates				
Term	Estimate	Std Error	t Ratio	Prob> t
Intercept	0.082	0.001483	55.28	<.0001*
NCS (equivalents)(1.04,1.07)	-0.00625	0.001658	-3.77	0.0130*
Temperature (C)(13,21)	0.00625	0.001658	3.77	0.0130*
Addition Rate (min.)(30,75)	-0.00375	0.001658	-2.26	0.0732
Sulfuric acid (mol%)(0.8,1.2)	-0.00625	0.001658	-3.77	0.0130*

FIG.8E

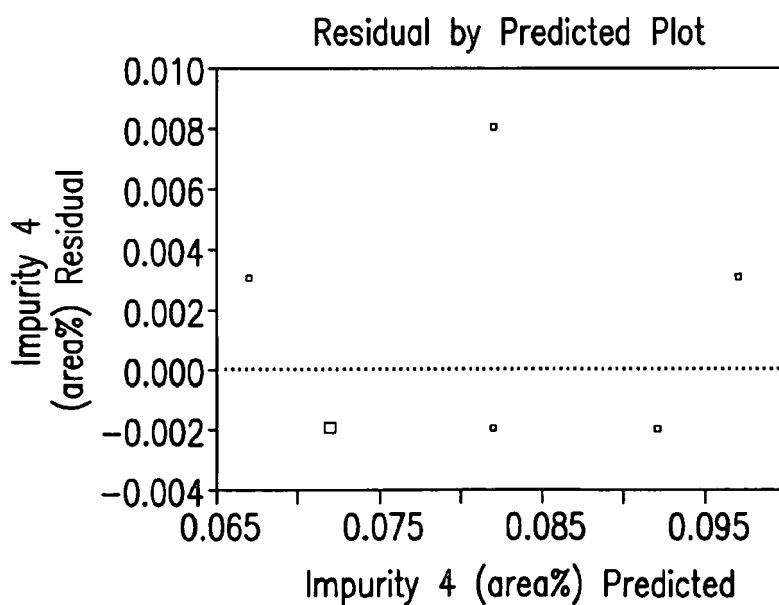


FIG.8F

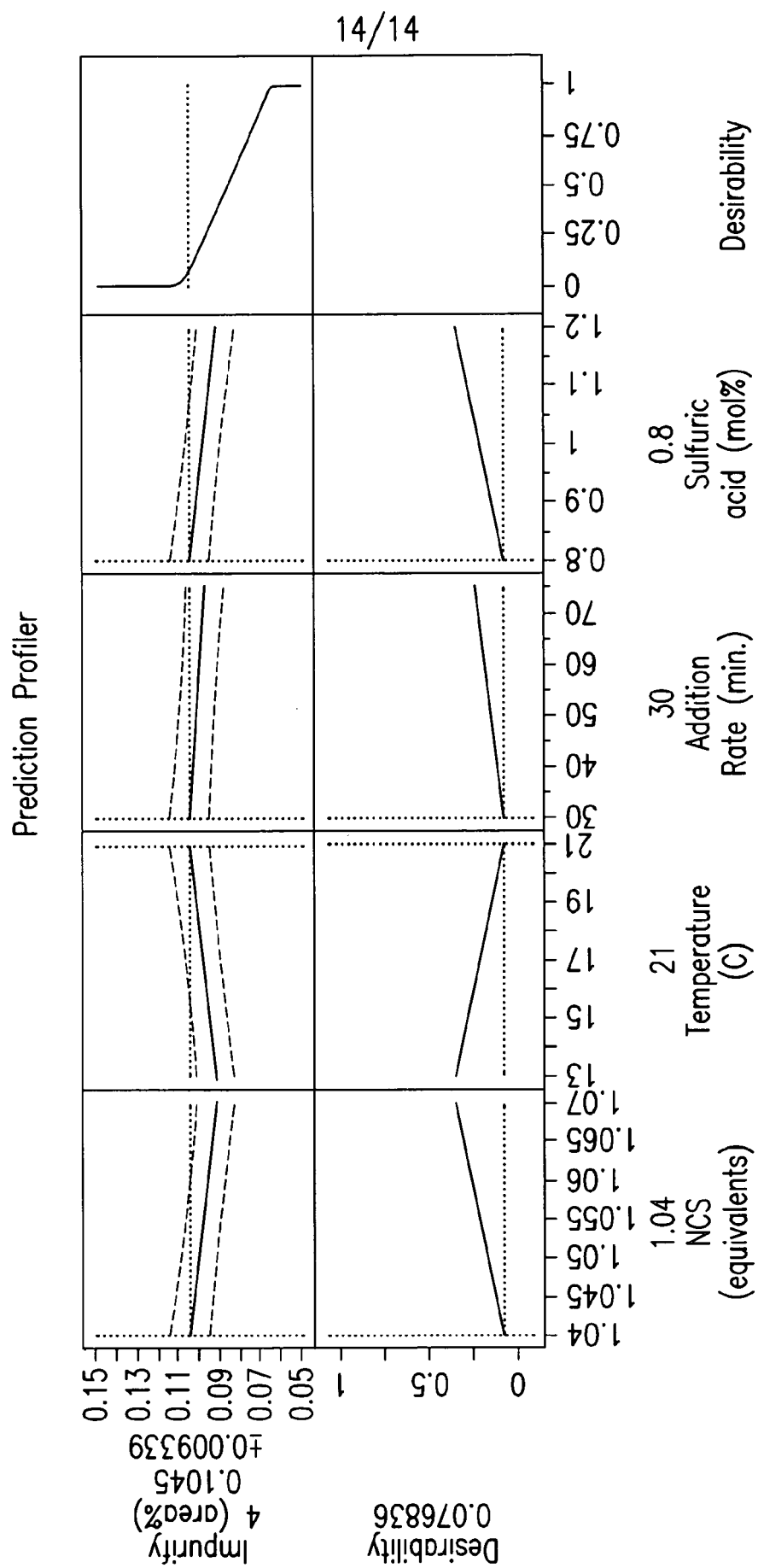


FIG. 8G