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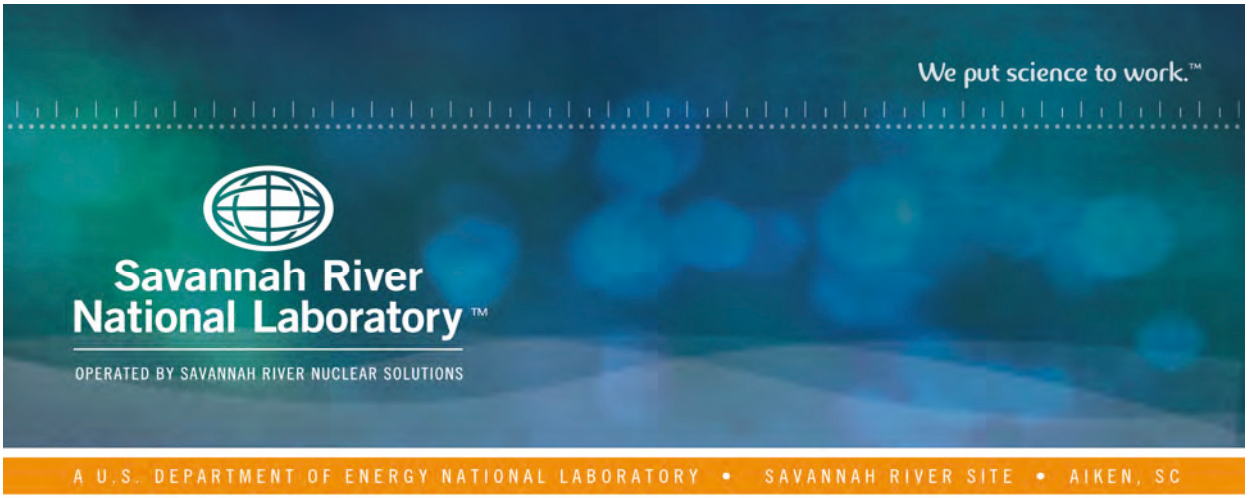

Defense Waste Processing Facility Nitric-Glycolic Flowsheet Chemical Process Cell Chemistry: Part 2

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July 2020

SRNL-STI-2017-00172, Revision 1

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Prepared for
U.S. Department of Energy

SRNL-STI-2017-00172
Revision 1

Keywords: *DWPF, Chemical Processing Cell, Glycolic Acid*

Retention: *Permanent*

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LIST OF REVISIONS		
Revision Number	**Summary of Changes**	**Date**
1	Corrected formula for nitrite destruction on page 58 and 66	7-27-2020

REVIEWS AND APPROVALS

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EXECUTIVE SUMMARY

The conversions of nitrite to nitrate, the destruction of glycolate, and the conversion of glycolate to formate and oxalate were modeled for the Nitric-Glycolic flowsheet using data from Chemical Process Cell (CPC) simulant runs conducted by Savannah River National Laboratory (SRNL) from 2011 to 2016. The goal of this work was to develop empirical correlation models to predict these values from measureable variables from the chemical process so that these quantities could be predicted a-priori from the sludge or simulant composition and measurable processing variables. The need for these predictions arises from the need to predict the REDuction/OXidation (REDOX) state of the glass from the Defense Waste Processing Facility (DWPF) melter. This report summarizes the work on these correlations based on the aforementioned data. Previous work on these correlations was documented in a technical report covering data from 2011-2015.[1] This current report supersedes this previous report. Further refinement of the models as additional data are collected is recommended.

The glass REDOX depends on the concentrations of nitrate and manganese (oxidants), and of glycolate, formate, oxalate, carbon, and antifoam (reductants) in the melter feed. The waste sludge contains nitrite, nitrate, manganese (Mn), and oxalate. Virtually all of the nitrite is converted to nitrate or $NO+NO_2+N_2O$ gases in the CPC. The portion of the nitrite converted to nitrate increases the amount of nitrate in the sludge. The amount of glycolate in the final melter feed depends on the amount of the glycolic acid feed that is destroyed. Similarly, the amounts of formate and oxalate formed during the decomposition of glycolic acid are required.

The material balance on carbon was found to not close in most cases. Generally, there was less carbon at the end of testing compared to the inputs. The most uncertain product variable was glycolate, so material balances were performed where the glycolate concentration was adjusted, usually upward, to close the balance. Correlation versus the original, as-measured, data was generally poor, but correlation against the material balance adjusted values was greatly improved. It was also shown that the correlation of the measured REDOX versus the predicted REDOX was much better when the material balance adjusted glycolate values were used.

Three data series were primarily used during the regressions of the data; these series were 1) Sludge Batch 9 NG flowsheet simulant runs NG51-62 (SB9-NG); 2) Scaled Runs + Bounding Hydrogen Runs (SR+BH); and 3) Runs GN43-50 and 57 (43-50,57). The glycolate destruction was found to correlate with acid stoichiometry (AS), percent reducing acid (PRA), and for some data series, headspace to simulant volume ratio (HSV), mercury (Hg), and nitrate. Although glycolate destruction for pairs of data series (e.g., [SB9-NG] *and* [SR+BH]) were found to depend on HSV, the combination of all three data series was not found to have significant dependence on this variable. The best model for glycolate destruction depended on AS, nitrate, and Hg. This model predicted the product glycolate compositions of the data to within 92-106%.

The conversion of glycolate to formate was high when noble metals and Hg were not present, with values up to 100%. When noble metals and Hg were present, this conversion ranged from zero to 7%, and was dependent on AS. Lower AS gave higher conversions to formate.

The conversion to oxalate was found to depend on the AS and the initial concentration of nitrite. An alternative fit versus AS and the form of ruthenium (Ru) used is a possible alternative. This fit was somewhat less statistically significant. This second model predicts that more oxalate is formed when Ru-nitrosyl nitrate is used rather than Ru chloride. The conversion of glycolate to oxalate ranged from zero to 6%.

The conversion of nitrite to nitrate depended primarily on AS and PRA, with HSV and Hg being significant when these variables were varied. For multiple series of data, nitrite was also needed to

distinguish between data series, and the effect of HSV became insignificant. The best model for nitrite to nitrate conversion depended on AS, PRA, nitrite, and Hg.

The 95% confidence intervals on the predicted values of glycolate destruction, glycolate to oxalate conversion, and nitrite to nitrate conversion were used to determine the uncertainty in the predicted REDOX when starting with only the composition of the sludge, AS, and PRA. Using the 95% confidences on an individual value (that is the confidence in getting a particular value for one single test as opposed to what the mean would be for multiple tests), the uncertainty in the predicted REDOX was calculated. The uncertainty in the actual product composition glycolate, oxalate, formate, and nitrate concentrations translated to an uncertainty in the REDOX value of ±0.1,which is approximately the uncertainty claimed in the REDOX model itself.

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43-50,57	Nitric-Glycolic Flowsheet Simulant Runs GN43-50 and GN57
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AS	Acid Stoichiometry
BH	Bounding Hydrogen Nitric-Glycolic Flowsheet Simulant Runs GN80-83
CEF	Cold Cap Evaluation Furnace
CPC	(DWPF) Chemical Process Cell
CQ	Caustic Quench (IC Method)
CS	Calcine Solids
CSBal	Calcine Solids Balance
DWPF	Defense Waste Processing Facility
ELN	Electronic Laboratory Notebook
FAVC	Formic Acid Vent Condenser
FTIR	Fourier Transform Infrared Spectroscopy Or Spectrometer
GF	Glycolic-Formic
GN	Nitric-Glycolic
G_D	Glycolate Destruction (%)
GtoOx	Glycolate to Oxalate Conversion (%)
GtoF	Glycolate to Formate Conversion (%)
HSV	Headspace Volume to Sludge Volume
IC	Ion Chromatography
JMP	JMP Statistical Software Package
KMA	Koopman Minimum Acid
MBal	Material Balance
MS	Mass Spectrometer or Spectrometry
MWWT	Mercury Water Wash Tank
NA	Not Available
N_C	Nitrite to Nitrate Conversion (%)
NG	Nitric-Glycolic
NM	Noble Metals (& Hg) Present

LIST OF ABBREVIATIONS AND DEFINITIONS (continued)

No NM	No NM or Hg Present Nitric-Glycolic Flowsheet Simulant Runs GN51-56, GN58-59
OG	Offgas
PRA	Percent Reducing Acid
REDOX	Reduction Oxidation
SASV	Headspace Surface Area to Sludge Volume
SB	Sludge Batch
SB6	Sludge Batch 6
SB6D	Sludge Simulant for SB6 Tank 40 Based on August 2009 Composition Projection
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SB9-NF	SB9 Nitric-Formic Flowsheet Simulant Runs
SB9-NG	SB9 Nitric-Glycolic Flowsheet Simulant Runs NG51-62
SC	Shielded Cells
SC-18	Shielded Cells Run SC-18
Simulant	Non-Radioactive Simulant for a Radioactive Waste Sludge
SME	Slurry Mix Evaporator
SMECT	Slurry Mix Evaporator Condensate Tank
SR	Scaled Runs Nitric-Glycolic Flowsheet Simulant Runs GN70-79
SR+BH	Scaled Runs Nitric-Glycolic Flowsheet Simulant Runs GN70-79 & Bounding Hydrogen Nitric-Glycolic Flowsheet Simulant Runs GN80-83
SRAT	Sludge Receipt and Adjustment Tank
SRAT/SME	Both SRAT and SME Cycles Performed
SRAT (SME)	SRAT Cycle Only with Frit Added (No SME Cycle)
SRNL	Savannah River National Laboratory
TIC	Total Inorganic Carbon
TS	Total Solids
WAPS	Waste Acceptance Product Specifications
wt %	Weight Percent

1.0 Introduction

Development of models for glycolate destruction, glycolate to oxalate conversion, glycolate to formate conversion, and nitrite to nitrate conversion for the nitric-glycolic (NG) flowsheet were performed as requested by the Defense Waste Processing Facility (DWPF) Technical Task Request[2] and as described in the Task Technical and Quality Assurance Plan (SRNL-RP-2014-01183, Rev. 0 Task Activity 4.2).[3] A memorandum that provided additional details on the work to be done was also issued.[4]

To implement the Nitric-Glycolic (NG) flowsheet in DWPF, adjustment of the Chemical Processing Cell (CPC) acid stoichiometry (AS) and of the reducing/oxidizing (glycolic/nitric) acid ratio to achieve a desired glass REDuction/OXidation (REDOX) must be possible using only the incoming composition of the sludge. Historically, the nitric-glycolic experimental data have been abbreviated 'GN', 'NG', or 'GF' (GF meant glycolic-formic (with nitric) when glycolic/formic mixtures were used), and was carried over into the initial glycolic only with nitric tests. Glass REDOX is the balance of reducing species to oxidizing species in the glass, e.g., Fe^{2+} vs. Fe^{3+}. The REDOX of the glass controls the deposition of metals that can short the joule-heating electrodes (too reducing) or cause foaming and increase the volatility of radionuclides such as Tc, Ru, and Cs (too oxidizing). The glass REDOX is dependent on the nitrate, glycolate, and several other concentrations. The final nitrate and glycolate concentrations are dependent on the nitrite to nitrate conversion and the glycolate destruction percentages, which are not necessarily known a priori. In this work, data from 56 simulant tests and one real waste test were analyzed and these percentages were regressed versus sludge composition variables, equipment configuration variables, and processing parameters that might be expected to affect these percentages. This report describes this work and proposes empirical correlations (models) for nitrite to nitrate conversion, glycolate destruction, and glycolate to formate conversion. Recommendations for future work on these correlations and on the values input to them are made.

2.0 Experimental Data

2.1 Data Series

The data used for this study were generated from multiple simulant CPC tests that occurred from 2011 to 2016. Some tests were complete SRAT/SME (Sludge Receipt and Adjustment Tank / Slurry Mix Evaporator) cycles, some were only SRAT cycles, and some were SRAT cycles with frit added without performing an actual SME cycle. The early Alternate Reductant tests with both glycolic and formic acids were excluded from consideration because DWPF will not operate with mixed acids except during the short transition period between the flowsheets. The applicability of any particular run was qualitatively judged by considering the values for the nitrate and glycolate anion product concentrations and percent conversions during the test. Values judged to be unrealistic were excluded. For some older samples, as available, the slurry was reanalyzed for anions using the "caustic quench" (CQ) method.[5] The data series used are described in Table 1; the symbols used on plots, the color key used in tables, the 'shorthand names' for the data series, and the number of data points are given. The key given by Table 1 will be used throughout this report.

Table 1. Description of Data Series

Symbol	Table Shading	Data Set	Shorthand Name	Number of Data Points
●		GN34-38 (originally GF34-38)	30s	10
■		GN40-41	40-41	2
▲		GN43-50	43-50	8
□		GN57 (with noble metals & Hg)	57	1
▲&□		GN43-50 & 57	43-50,57	9
▲		GN51-56,58-59 No noble metals or Hg	No NM	8
◆		GN70-79 Scaled Runs (see note below)	SR	10
◆		GN70-75 Scaled Runs (see note below)	SR	6
◇		GN76-79 Scaled Runs (22-L & 220-L Runs)		4
◆		GN80-83 Bounding H_2	BH	4
■		SB9-NG51-62	SB9-NG	12
◆		Shielded Cells Run SC-18	SC-18	1

Note: When the 22-L & 220-L runs (GN76-79) are shown separately from the 4-L runs (GN70-75), the GN70-75 runs are shown by ◆ and the GN76-79 runs are shown by ◇. When these runs are not shown with different symbols, all (GN70-79) are shown by ◆.

The descriptions of the simulant run data used are shown in Table 2. The oldest data series generally had less reliable data. Some of the values in Table 2 are slightly different than the Part 1 report. Further delving into the input data showed that some values that had been used were slightly incorrect.

Table 2. Description of Data Used for Models

Run Number	Cycles	Sludge Type	Scale (vessel volume L)	Acid Stoich (KMA)	Percent Reducing Acid	Noble Metals & Mercury	Run Series Description
GN34	SRAT/SME	HiFeHiMn	4	104.0%	63.1%	Y	**Matrix Study** at High and Low Mn and Fe Concentrations
GN34b	SRAT	HiFeHiMn	4	103.5%	63.0%	Y	
GN34c	SRAT	HiFeHiMn	4	104.0%	63.1%	Y	
GN35	SRAT/SME	LoFeHiMn	4	100.0%	56.9%	Y	
GN36	SRAT/SME	HiFeLoMn	4	106.1%	59.3%	Y	
GN36b	SRAT	HiFeLoMn	4	106.1%	59.3%	Y	
GN36c	SRAT	HiFeLoMn	4	106.1%	59.3%	Y	
GN37	SRAT/SME	LoFeLoMn	4	100.0%	60.4%	Y	
GN37b	SRAT	LoFeLoMn	4	100.0%	60.3%	Y	
GN38	SRAT	LoFeLoMn	4	125.0%	59.0%	Y	
GN40	SRAT/SME	2.0 M Na SB8-B	4	134.0%	53.7%	Y	**Less Washed Sludge** (SB8 type sludge)
GN41	SRAT/SME	2.5 M Na SB8-B	4	130.0%	53.7%	Y	
GN43	SRAT	SB6J	4	105.0%	51.9%	Y	**Acid Stoichiometry Tests** (Blend of SB6 sludges used)
GN44	SRAT	SB6J	4	85.0%	53.9%	Y	
GN45	SRAT	SB6J	4	95.0%	58.2%	Y	
GN46	SRAT	SB6J	4	90.0%	58.9%	Y	
GN47	SRAT	SB6J	4	100.0%	56.4%	Y	
GN48	SRAT	SB6J	4	100.0%	61.9%	Y	
GN49	SRAT	SB6J	4	100.0%	50.8%	Y	
GN50	SRAT	SB6J	4	100.0%	45.3%	Y	
GN51	SRAT (SME)	SB6I	4	100.0%	54.6%	N	**Tests Supporting CEF Runs**
GN52	SRAT (SME)	SB6I	4	100.0%	55.1%	N	
GN53	SRAT (SME)	SB6I	4	125.0%	52.5%	N	
GN54	SRAT (SME)	SB6I	4	100.0%	51.0%	N	
GN55	SRAT (SME)	SB6I	4	100.0%	47.7%	N	
GN56	SRAT (SME)	SB6I	4	100.0%	49.4%	N	
GN57	SRAT (SME)	SB6I	4	110.0%	49.0%	Y	Approximate comparison to GN56 with Noble Metals & Hg added
GN58	SRAT (SME)	SB6I	4	125.0%	47.7%	N	**Tests Supporting CEF Runs**
GN59	SRAT (SME)	SB6I	4	125.0%	46.0%	N	
GN70	SRAT/SME	SB8-E	4	100.0%	58.3%	Y	**Scaled Runs** (mix of the 'B' and 'C' sludges to approximate SB8)
GN71	SRAT/SME	SB8-E	4	125.0%	55.0%	Y	
GN72	SRAT/SME	SB8-E	4	100.0%	52.1%	Y	
GN73	SRAT/SME	SB8-E	4	110.0%	52.2%	Y	
GN74	SRAT/SME	SB8-E	4	100.0%	54.5%	Y	
GN75	SRAT/SME	SB8-E	4	110.0%	52.2%	Y	
GN76	SRAT/SME	SB8-E	22	100.0%	58.3%	Y	
GN77	SRAT/SME	SB8-E	22	110.0%	52.2%	Y	
GN78	SRAT/SME	SB8-E	220	110.0%	52.2%	Y	
GN79	SRAT/SME	SB8-E	220	100.0%	54.5%	Y	
GN80	SRAT/SME	SB8-E	4	110.0%	54.2%	Y	**Bounding Hydrogen**
GN81	SRAT/SME	SB8-E	4	110.0%	54.2%	Y	
GN82	SRAT/SME	SB8-E	4	110.0%	54.2%	Y	
GN83	SRAT/SME	SB8-E	4	110.0%	54.2%	Y	
NG51	SRAT	SB9	4	83.7%	54.1%	Y	**SB9 Flowsheet Development**
NG52	SRAT	SB9	4	116.0%	62.3%	Y	
NG53	SRAT	SB9	4	83.6%	62.6%	Y	
NG54	SRAT	SB9	4	117.0%	54.1%	Y	
NG55	SRAT	SB9	4	100.0%	58.3%	Y	
NG55A	SRAT	SB9	4	100.0%	58.4%	Y	
NG56	SRAT	SB9	4	100.0%	52.3%	Y	
NG57	SRAT	SB9	4	99.9%	64.4%	Y	
NG58	SRAT	SB9	4	76.9%	58.4%	Y	
NG59	SRAT	SB9	4	123.0%	58.4%	Y	
NG60	SRAT/SME	SB9	4	100.0%	54.1%	Y	
NG61	SRAT/SME	SB9	4	100.0%	54.1%	Y	
NG62	SRAT/SME	SB9	4	97.4%	55.7%	Y	
SC-18	SRAT/SME	SB9	4	78.0%	57.4%	Y	**Shielded Cells Run with SB9-A Tank 40**

(see Table 1 for color key)

The 30s series was performed using four simulant compositions: 1) high Fe, high Mn; 2) high Fe, low Mn; 3) low Fe, high Mn; and 4) low Fe, low Mn. The specific concentrations of these simulants are given in Table 5. These simulants were not designed to represent a real waste tank or sludge batch composition.[6] These runs have been included in the analysis because they were performed with uncharacteristic Fe and Mn concentrations and so provided more variation in these concentrations for the modeling. Note that in the original data, these runs were referred to as 'GF' runs, even though they did not use formic acid.

The data for the 30s series are the least reliable because these samples were approximately three years old and were not originally analyzed using the CQ anions method. All of the available 30s series samples were reanalyzed by the CQ method in 2015. However, some samples appeared to have lost glycolate while gaining oxalate, so these data are more uncertain. Likewise, the CQ method gives higher oxalate, so it is unclear how much of the increase in the measured oxalate was from the better method and how much was from actual increases in concentration over time.

Series 40-41 was unusual in that the simulant compositions mimicked less-washed sludges with higher sodium concentrations and significantly higher oxalate concentrations. These simulant compositions approximated Sludge Batch 8 had it been less washed.

The 43-50 series was performed to study the effects of AS and PRA. The simulant used was a blend of several SB6 simulants (SB6D, SB6E, SB6H) and was called SB6J.

The No NM series was done with no noble metals or Hg present to develop a simulant to be made by an offsite vendor for large-scale melter testing. Run 57 used the same simulant but was done with noble metals (NM) and mercury (Hg), so was not generally included with the No NM data, except for a few comparisons. This run was the only run with noble metals and Hg that was not part of a series of runs at a specific simulant composition. This simulant was also unusual because it had an uncharacteristically low nitrite to nitrate ratio. Simulant nitrite to nitrate ratios are typically about 1.5-1.9 to mimic actual waste tank compositions. The GN51-59 simulant had a nitrite to nitrate ratio of 0.90. This simulant was based on simulant batch SB6I that was used for the Cold-Cap Evaluation Furnace (CEF) melter testing with the NG flowsheet.[7]

Runs GN70-79 were called the Scaled Runs (SR) because tests were run at nominally 4-L, 22-L, and 220-L scales. These runs used the simulant SB8-E that was made from components B and C of the "ABC" simulants[8] and additional trim chemicals.[9] This simulant approximated the SB8 simulant used for the final SB8 flowsheet testing.[10] This same simulant was used for the Bounding Hydrogen series (BH).[11] The most significant difference between the simulants for SR and BH was the concentration of Hg.

The composition of the SB9-NG simulant approximated the expected SB9 Tank 40 composition. These runs were designated SB9-NG-51 through SB9-NG-62. Formic acid flowsheet tests were also performed using this simulant composition (SB9-NF). The SB9-NG runs were the simulant runs supporting the Shielded Cells Run SC-18 (SC-18).

The destruction of glycolate and the conversions of glycolate to oxalate and formate and of nitrite to nitrate were calculated from the starting concentrations, amounts of acids added, and the final SRAT or SME anion concentrations. The acid calculations were used to calculate these conversions, and a spreadsheet was developed for data analysis that also calculated the conversions. When available, the SRAT product compositions were used rather than the SME product compositions. It was difficult to close material balances using some of the SME data because of the increased uncertainty in the actual amount of initial simulant in the SME product due to uncertainties in the mass and composition of samples removed, incomplete product removal and weighing, and errors in the frit to simulant ratio used for SME products based on calculated SRAT product masses.

2.2 Adjustments to Data

To improve the accuracy of the material balances, a balance on the calcine solids (CS) of the simulant amount used was performed. If no samples are removed, the amount of calcine solids from the simulant in the SRAT or SME product should be the same as the simulant initially added to the SRAT since neither nitric acid nor glycolic acid adds to the CS value. This CS balance (CSBal) was done on most of the runs. The result of the balance is an updated final SRAT mass upon which the material balances on carbon and nitrogen can be performed. In many runs, especially with the SB9-NG series, the CSBal SRAT mass was higher than that calculated by the standard accounting method for mass in the acid calculations. For other runs, the CSBal gave different final SRAT masses, but these masses were not greatly different than the original calculations. The CSBal method was applied to the SB9-NG, 43-50, 57, and No NM series, and the GN34 run. Collectively, both the data adjusted by and not adjusted by the CSBal method are designated 'Measured'; if adjusted, the CSBal values are used, and if not adjusted the original values are used. All of the acid calculations used and data analysis spreadsheets are recorded in Experiment T7909-00035-16 in the Electronic Laboratory Notebook (ELN).

From the anion analyses, the starting anion concentrations, the amounts of nitric and glycolic acid added, the conversion of nitrite to nitrate and the destruction of glycolate were calculated using the acid calculation spreadsheet for each run. The conversions of glycolate to formate and to oxalate were also calculated.

The material balances on carbon and nitrogen were closed by adjusting the final concentrations in the SRAT of glycolate and nitrate, respectively. The material balance species are summarized in Table 3. It was assumed that there was no formate in the feeds (although there could be small quantities of formate in the glycolic acid), that there was no nitrite remaining in the simulant, that there was no nitrite in the final simulant, and that no other significant offgases were generated (CO, N_2, NH_3).

It is noted that the feed simulant carbonate, or Total Inorganic Carbon (TIC) value can be very inaccurate. The best values found from reports or analytical results sheets were used, but it should be realized that these values could be off by up to 20%. The concentrations of other anions by Ion Chromatography (IC) are generally considered to be ±10%, but are generally more accurate. However, there does still appear to be a low bias in the glycolate values measured by the CQ anions method.

Table 3. Material Balance Quantities

Species	Feeds	Products
Carbon	Carbonate (TIC) Oxalate Glycolic Acid	Glycolate Oxalate Formate CO_2
Nitrogen	Nitrite Nitrate Nitric Acid	Nitrate in Simulant Nitrogen Oxides (NO, NO_2, N_2O) at Offgas Exit Nitrite and Nitrate in Condensates (Dewater, SMECT, MWWT, FAVC)

SMECT: Slurry Mix Evaporator Condensate Tank; MWWT: Mercury Water Wash Tank;
FAVC: Formic Acid Vent Condenser

The offgas CO_2 was measured by gas chromatography (GC) in all runs except the No NM series. Carbon dioxide was also measured by mass spectrometry (MS) in the SR, BH, and SB9-NG series, and by Fourier Transform Infrared Spectroscopy (FTIR) in some runs in these series. The gases NO and NO_2 were also measured in the SR, BH, and SB9-NG series by MS, and by FTIR in some runs. The total summation of each gas was calculated from the total offgas flow that was calculated from helium dilution. (Incorrect mass flow meter readings present in older run data were corrected.) Where duplicate values were available (e.g., CO_2 from GC, MS, FTIR), the agreement was generally good and the largest value

was used. The concentrations of nitrite and nitrate in the offgas condensate samples were measured by Ion Chromatography (IC).

In the Material Balance (MBal) method, it was assumed that the offgas data were accurate and that the uncertainty was contained in the SRAT or SME product analyses. Accordingly, the product glycolate and nitrate values were adjusted to close the material balances to 100%. Adjustment of the oxalate and formate values could also have been done. The analysis for formate has been proven to be accurate, but the oxalate values may be biased low. The decision was made to only adjust the glycolate values given no definitive information otherwise. No material balance on nitrogen could be done for runs where nitrogen species in the offgas and offgas condensate were not measured. Carbon balances could be done for all runs except the No NM series and Run 57 where there either were no CO_2 measurements or they were missing. For the No NM series and Run 57, to approximate the effect of a material balance on the glycolate concentration, generation of 0.550 mol of CO_2 was assumed.

The percent conversions were empirically correlated to the variables shown in Table 4 using the JMP Pro Version 11.2.1 statistical software.[12] These variables were chosen because they were hypothesized to have an effect on the conversion of nitrite to nitrate or the destruction of glycolate. Acid stoichiometry and PRA are obvious choices. The concentrations of nitrite and nitrate could affect the nitrite conversion; the nitrite to nitrate conversion could affect the glycolate destruction if nitrite or nitrous acid, or the NO_x generated, is involved in destruction reactions. Oxalate does not seem a likely variable except that it is a glycolate destruction product and might thus affect the formation of oxalate. Manganese and Hg are both involved in REDOX reactions that can destroy glycolic acid and possibly nitrous acid. Iron (Fe) was added because of its high concentration and that it is slightly dissolved by glycolic acid; it was reasoned that if a small amount of a large species reacted, it could affect the chemistry noticeably. Headspace Surface Area to Sludge Volume (SASV) and Headspace Volume to Sludge Volume (HSV) were added for reasons described in Section 2.4.

There are many potential additional differences between runs that could account for differences in the results that would be hard to quantify. Some differences include the addition rate of the acids, the down time between nitric acid and glycolic acid additions, the time between glycolic acid addition and achieving boiling, the length of boiling during the SRAT cycle, the boilup rates used, etc. So many possible differences could be added as variables that there would be too many variables to perform reliable statistical analysis. Also, some of these potential variables would not be known a priori. Therefore, the uncertainty in the model predictions will include the effect of such planned and unplanned differences.

Table 4. Empirical Variables for Models

Empirical Variable	Abbreviation
Acid Stoichiometry (%)	AS
Percent Reducing Acid (%)	PRA
Initial Nitrite Concentration (mg/kg)	Nitrite
Initial Nitrate Concentration (mg/kg)	Nitrate
Initial Oxalate Concentration (mg/kg)	Oxalate
Iron Concentration (wt % CS)	Fe
Manganese Concentration (wt % CS)	Mn
Mercury (wt % TS)	Hg
Headspace Surface Area to Sludge Volume Ratio (cm^{-1})	SASV
Headspace Volume to Sludge Volume Ratio	HSV

Note that the AS was defined using the Koopman minimum acid (KMA) stoichiometry[13] equations for the NF flowsheet with glycolic acid substituted directly for formic acid on a molar basis. The actual correct minimum acid calculation for the NG flowsheet is not known, but using the KMA values serves as a way

to compare runs. In the future, the new AS values can be substituted for the KMA values that are tabulated herein. The variables considered are given in Table 5.

Acid stoichiometry is calculated for each simulant composition and depends on the base equivalents, carbonate, nitrite, Hg, Mn, Ca and Mg for the KMA AS equation. Since the AS depends on several of the concentrations that are used as variables for fitting data (nitrite, Mn, Hg), AS is therefore confounded with these variables and difficulty in distinguishing the effects of AS from these variables would be expected in the empirical models.

The noble metals, Hg, and Mn concentrations are given in Table 6. The concentration ranges of noble metals in the Tank 40 Waste Acceptance Product Specifications (WAPS) samples for sludge batches back to SB4 are also shown for reference.[14] Note that the amounts of Pd, Rh, and Ru used in the 30s series and 43-50,57 series were significantly higher than in the later SR+BH and SB9-NG series or in real waste. Also note that the form of Ru used in the SB9-NG series was different than in the other simulant series. Noble metals play a role in the chemistry, especially with the destruction of formate generated during the decomposition of glycolic acid, and appear to also affect the generation of oxalate. The noble metals concentrations used were not significantly varied in the series with the most reliable data (SR, BH, SB9-NG), so noble metals concentrations were not used as variables except for the glycolate to oxalate conversion. For the glycolate to formate conversion, the presence or absence of noble metals and Hg was used to distinguish data series.

Table 5. Variables Used in the Regressions

Run Number	Scale (vessel volume L)	Surface Area to Sludge Volume Ratio (cm^{-1})	Head Space Volume to Sludge Volume Ratio	Acid Stoich (KMA)	Percent Reducing Acid	Noble Metals & Mercury	Initial Nitrite, mg/kg	Initial Nitrate, mg/kg	Initial Oxalate, mg/kg	Mn wt% CS	Fe wt% CS
GN34	4	0.522	1.194	104.0%	63.1%	Y	17,900	13,600	300	4.04	32.4
GN34b	4	0.522	1.194	103.5%	63.0%	Y	17,900	13,600	300	4.04	32.4
GN34c	4	0.522	1.194	104.0%	63.1%	Y	17,900	13,600	300	4.04	32.4
GN35	4	0.523	1.194	100.0%	56.9%	Y	9,610	5,880	7,220	5.12	19.7
GN36	4	0.522	1.194	106.1%	59.3%	Y	17,800	13,400	275	0.69	31.5
GN36b	4	0.522	1.194	106.1%	59.3%	Y	17,800	13,400	275	0.69	31.5
GN36c	4	0.522	1.194	106.1%	59.3%	Y	17,800	13,400	275	0.69	31.5
GN37	4	0.522	1.194	100.0%	60.4%	Y	18,100	13,300	295	0.66	12.2
GN37b	4	0.518	1.176	100.0%	60.3%	Y	18,100	13,300	295	0.66	12.2
GN38	4	0.518	1.170	125.0%	59.0%	Y	18,100	13,300	295	0.66	12.2
GN40	4	0.490	1.106	134.0%	53.7%	Y	13,500	7,900	18,800	4.63	13.7
GN41	4	0.490	1.106	130.0%	53.7%	Y	15,800	9,940	20,000	4.23	12.4
GN43	4	0.576	1.336	105.0%	51.9%	Y	12,100	6,700	387	7.09	17.8
GN44	4	0.576	1.336	85.0%	53.9%	Y	12,100	6,700	387	7.09	17.8
GN45	4	0.576	1.336	95.0%	58.2%	Y	12,100	6,700	387	7.09	17.8
GN46	4	0.577	1.336	90.0%	58.9%	Y	12,100	6,700	387	7.09	17.8
GN47	4	0.456	1.015	100.0%	56.4%	Y	12,100	6,700	387	7.09	17.8
GN48	4	0.456	1.015	100.0%	61.9%	Y	12,100	6,700	387	7.09	17.8
GN49	4	0.456	1.015	100.0%	50.8%	Y	12,100	6,700	387	7.09	17.8
GN50	4	0.456	1.015	100.0%	45.3%	Y	12,100	6,700	387	7.09	17.8
GN51	4	0.471	1.055	100.0%	54.6%	N	9,540	10,600	781	7.08	22.2
GN52	4	0.471	1.055	100.0%	55.1%	N	9,540	10,600	781	7.08	22.2
GN53	4	0.471	1.055	125.0%	52.5%	N	9,540	10,600	781	7.08	22.2
GN54	4	0.471	1.055	100.0%	51.0%	N	9,540	10,600	781	7.08	22.2
GN55	4	0.471	1.055	100.0%	47.7%	N	9,540	10,600	781	7.08	22.2
GN56	4	0.471	1.055	100.0%	49.4%	N	9,540	10,600	781	7.08	22.2
GN57	4	0.468	1.042	110.0%	49.0%	Y	9,540	10,600	781	7.08	22.2
GN58	4	0.471	1.055	125.0%	47.7%	N	9,540	10,600	781	7.08	22.2
GN59	4	0.471	1.055	125.0%	46.0%	N	9,540	10,600	781	7.08	22.2
GN70	4	0.454	1.011	100.0%	58.3%	Y	12,400	8,100	2,010	7.56	24.4
GN71	4	0.454	1.011	125.0%	55.0%	Y	12,400	8,100	2,010	7.56	24.4
GN72	4	0.454	1.011	100.0%	52.1%	Y	12,400	8,100	2,010	7.56	24.4
GN73	4	0.454	1.011	110.0%	52.2%	Y	12,400	8,100	2,010	7.56	24.4
GN74	4	0.454	1.011	100.0%	54.5%	Y	12,400	8,100	2,010	7.56	24.4
GN75	4	0.454	1.011	110.0%	52.2%	Y	12,400	8,100	2,010	7.56	24.4
GN76	22	0.094	0.395	100.0%	58.3%	Y	12,400	8,100	2,010	7.56	24.4
GN77	22	0.120	0.547	110.0%	52.2%	Y	12,400	8,100	2,010	7.56	24.4
GN78	220	0.108	0.887	110.0%	52.2%	Y	12,400	8,100	2,010	7.56	24.4
GN79	220	0.082	0.616	100.0%	54.5%	Y	12,400	8,100	2,010	7.56	24.4
GN80	4	0.454	1.010	110.0%	54.2%	Y	12,400	8,100	2,010	7.56	24.4
GN81	4	0.454	1.010	110.0%	54.2%	Y	12,400	8,100	2,010	7.56	24.4
GN82	4	0.454	1.010	110.0%	54.2%	Y	12,400	8,100	2,010	7.56	24.4
GN83	4	0.454	1.010	110.0%	54.2%	Y	12,400	8,100	2,010	7.56	24.4
NG51	4	0.343	0.644	83.7%	54.1%	Y	10,200	5,730	3,510	8.74	24.5
NG52	4	0.343	0.644	116.0%	62.3%	Y	10,200	5,730	3,510	8.74	24.5
NG53	4	0.343	0.644	83.6%	62.6%	Y	10,200	5,730	3,510	8.74	24.5
NG54	4	0.343	0.644	117.0%	54.1%	Y	10,200	5,730	3,510	8.74	24.5
NG55	4	0.343	0.644	100.0%	58.3%	Y	10,200	5,730	3,510	8.74	24.5
NG55A	4	0.343	0.644	100.0%	58.4%	Y	10,200	5,730	3,510	8.74	24.5
NG56	4	0.343	0.644	100.0%	52.3%	Y	10,200	5,730	3,510	8.74	24.5
NG57	4	0.343	0.644	99.9%	64.4%	Y	10,200	5,730	3,510	8.74	24.5
NG58	4	0.343	0.644	76.9%	58.4%	Y	10,200	5,730	3,510	8.74	24.5
NG59	4	0.343	0.644	123.0%	58.4%	Y	10,200	5,730	3,510	8.74	24.5
NG60	4	0.343	0.644	100.0%	54.1%	Y	10,200	5,730	3,510	8.74	24.5
NG61	4	0.343	0.644	100.0%	54.1%	Y	10,200	5,730	3,510	8.74	24.5
NG62	4	0.310	0.560	97.4%	55.7%	Y	10,200	5,730	3,510	8.74	24.5
SC-18	4	0.340	0.630	78.0%	57.4%	Y	13,700	7,610	2,610	7.12	21.6

(see Table 1 for color key)

Table 6. Noble Metals, Hg, and Mn Concentrations

	Concentration (wt % TS)						
Runs:	**30s**	**43-50, 57**	**SR**	**BH**	**SB9-NG**	**SC-18**	**Tank 40 WAPS SB4-SB8 Range**
Ag	0.00140	0.00140	0.0164	0.0175	0.0139	0.0118	0.0099-0.0180
Pd	0.0790	0.0790	0.00338	0.0988	0.00370	0.00251	0.00125-0.00317
Rh	0.0380	0.0380	0.0175	0.0475	0.0156	0.0124	0.0084-0.0207
Ru*	0.217	0.217	0.0830	0.2713	0.0762	0.0566	0.031-0.102
Hg	1.50	1.50	2.14	1.00	2.48	2.18	NA
	Concentration (wt % CS)						
Mn	varied	7.09	7.59	7.59	8.74	7.12	NA
	Concentrations Normalized on Lowest Value						
Ag	1.00	1.00	11.7	12.5	9.93	9.93	NA
Pd	0.8	0.8	0.03	1.00	0.04	1.10	NA
Rh	3.06	3.06	1.41	3.83	1.26	1.00	NA
Ru	3.83	3.83	1.47	4.79	1.35	1.00	NA

* Ru in SB9-NG runs was $Ru(NO)(NO_3)_3$; all other simulant runs used $RuCl_3$

The concentrations of nitrate, glycolate, oxalate, and formate in the products are given in Table 7. Where it could be calculated, the nitrogen material balance closure is tabulated along with the material balance adjusted nitrate values. The ratio of the MBal to measured concentration is also shown. The corresponding quantities for the carbon balance and glycolate are also shown.

The average nitrogen material balance closure, for the runs with nitrogen offgas data, was 97.2%, with only one run less than 90% and two runs at about 110%. Because the material balance closure on nitrogen was so close to 100%, it was deemed that the nitrate values for the runs without material balance checks would for the most part be considered good values. Empty cells in the table indicate no data were available.

The material balance closure for carbon was not as quantitative as for nitrogen, and the closure was somewhat run dependent. The carbon material balance closure values are shown in Tables 7. The 43-50 series had the lowest closure values, so the product glycolate concentrations would have been increased the most in these runs. As will be shown in Section 3.4, the glycolate destruction values using the Measured data often could not be correlated well with any of the input variables. However, the MBal values could be better correlated against the expected variables, so it appears that the glycolate concentrations in many of the products were higher than measured. Table 7 shows that the glycolate values needed to be increased by up to 22% to close the material balance. The average MBal to measured percentage for each data group is also shown in these tables. The average ratio of MBal to measured concentrations ranged from 102-117%. If it is assumed that the measurement of glycolate is accurate to ±10% of the value, 32 of the 56 concentrations would be within this criterion. However, as in Section 3.4, this accuracy may be inadequate for modeling the destruction of glycolate. No nitrate concentration is shown for the SC-18 run because the SRAT and SME product nitrate values differed greatly and indicated significantly more nitrate in the SME than in the SRAT, which could not be correct.

The MBal glycolate values are plotted versus the measured values in Figure 1. The black line is the fit of the MBal values. This line is statistically significant meaning that the difference between the MBal and measured values is also statistically significant. This graph clearly shows that most of the data points lie above the y=x line. No correlation of the difference with any of the variables used for modeling was found.

Table 7. Comparison of Measured and Material Balance Calculated Nitrate and Glycolate, and Oxalate and Formate Concentrations in SRAT or SME Products

Run	Nitrogen Material Balance Closure	Measured Nitrate (mg/kg)	Material Balance Nitrate (mg/kg)	Material Balance / Measured Value	Carbon Material Balance Closure	Measured Glycolate (mg/kg)	Material Balance Glycolate (mg/kg)	Material Balance / Measured Value	Oxalate (mg/kg)	Formate (mg/kg)
GN34	NA	69000	NA	NA	91%	58900	66000	112%	2070	0
GN34b	NA	55200	NA	NA	88%	46600	54700	117%	6690	467
GN34c	NA	54100	NA	NA	92%	50600	56100	111%	7490	285
GN35	NA	44300	NA	NA	96%	38800	41300	106%	17200	686
GN36	NA	65300	NA	NA	96%	48200	50700	105%	9550	682
GN36b	NA	55500	NA	NA	100%	51800	51600	100%	5160	283
GN36c	NA	55300	NA	NA	100%	53600	53900	101%	6070	301
GN37	NA	54500	NA	NA	89%	42200	48800	116%	3230	0
GN37b	NA	53000	NA	NA	99%	50700	51500	102%	4610	263
GN38	NA	59300	NA	NA	101%	57400	57000	99%	4810	435
			30s Average:		**95%**			**107%**		
GN40	NA	64600	NA	NA	98%	55600	57700	104%	18100	0
GN41	NA	58800	NA	NA	100%	49600	49600	100%	19300	404
GN43	NA	64900	NA	NA	82%	42100	53000	126%	1440	790
GN44	NA	54100	NA	NA	89%	35400	39900	113%	5230	2040
GN45	NA	56100	NA	NA	88%	47500	55900	118%	2010	1040
GN46	NA	53500	NA	NA	98%	48300	48200	100%	5980	550
GN47	NA	54900	NA	NA	89%	45400	51500	113%	4550	251
GN48	NA	47600	NA	NA	88%	49400	56700	115%	5110	232
GN49	NA	61900	NA	NA	81%	36000	46900	130%	4040	176
GN50	NA	69900	NA	NA	86%	34900	41700	119%	3760	134
			GN43-50 Average:		**88%**			**117%**		
GN51	NA	65100	NA	NA	93%	47500	52400	110%	1070	5910
GN52	NA	64200	NA	NA	92%	47300	52700	111%	1380	5990
GN53	NA	77400	NA	NA	91%	60200	67800	113%	1680	2730
GN54	NA	68200	NA	NA	94%	44700	48700	109%	1610	6020
GN55	NA	74700	NA	NA	94%	44200	47800	108%	1650	6070
GN56	NA	68100	NA	NA	104%	44000	41500	94%	1100	9300
GN57	NA	77000	NA	NA	79%	47600	52600	111%	4830	247
GN58	NA	89500	NA	NA	90%	54900	61800	113%	1390	525
GN59	NA	91100	NA	NA	86%	50400	60800	121%	1490	615
			GN51-56, 58, 59 Average:		**93%**			**110%**		

(see Table 1 for color key)

Table 7 Continued: Comparison of Measured and Material Balance Calculated Nitrate and Glycolate, and Oxalate and Formate Concentrations in SRAT or SME Products

Run	Nitrogen Material Balance Closure	Measured Nitrate (mg/kg)	Material Balance Nitrate (mg/kg)	Material Balance / Measured Value	Carbon Material Balance Closure	Measured Glycolate (mg/kg)	Material Balance Glycolate (mg/kg)	Material Balance / Measured Value	Oxalate (mg/kg)	Formate (mg/kg)
GN70	98%	51100	52600	103%	101%	51200	50500	99%	3440	849
GN71	92%	61500	67500	110%	101%	61000	60200	99%	2360	608
GN72	96%	58500	61500	105%	97%	45600	47300	104%	2640	677
GN73	96%	62300	65500	105%	96%	49000	51700	106%	2150	444
GN74	99%	55900	56600	101%	95%	46200	49700	108%	2360	571
GN75	97%	63000	64800	103%	100%	50500	50300	100%	2790	456
GN76	95%	49200	52200	106%	100%	48900	49000	100%	3860	1050
GN77	93%	59700	64800	109%	99%	49600	50400	102%	1270	474
GN78	94%	58900	63500	108%	99%	49600	50200	101%	2390	161
GN79	95%	52500	56000	107%	102%	48100	46700	97%	3450	313
			GN70-70 (SR) Average:		**99%**			**101%**		
GN80	NA	62100	NA	NA	97%	51300	53200	104%	2250	348
GN81	NA	63500	NA	NA	99%	52900	53600	101%	2460	356
GN82	NA	61100	NA	NA	95%	49600	53200	107%	2670	357
GN83	NA	63200	NA	NA	93%	49300	54200	110%	2800	388
			GN80-83 (BH) Average:		**96%**			**106%**		
			SR+BH Average:		**98%**			**103%**		
NG51	98%	56200	57900	103%	100%	45200	45300	100%	11400	847
NG52	100%	55000	55000	100%	97%	63100	65300	104%	7120	694
NG53	95%	46400	49200	106%	92%	47500	53200	112%	11200	1560
NG54	97%	66900	68900	103%	89%	54500	62900	115%	6300	374
NG55	98%	65700	67100	102%	89%	59500	69700	117%	10700	393
NG55A	110%	46000	41400	90%	96%	38400	40500	105%	6500	340
NG56	97%	68000	70400	104%	92%	49700	56000	113%	10700	348
NG57	101%	56900	56400	99%	100%	70700	70200	99%	10800	364
NG58	95%	50100	53000	106%	106%	47000	42700	91%	12800	1230
NG59	111%	48500	43200	89%	97%	44000	45700	104%	4700	574
NG60	93%	54900	58800	107%	93%	43700	47700	109%	7610	380
NG61	98%	56400	57700	102%	88%	39900	47900	120%	11100	262
NG62	87%	55000	64200	117%	87%	46500	56400	122%	6910	626
			NG51-62 Average:		**94%**			**109%**		
SC-18	NA	NA	NA	NA	98%	37100	38400	103%	4790	1450
			Average of All Tests:		**94%**			**108%**		

(see Table 1 for color key)

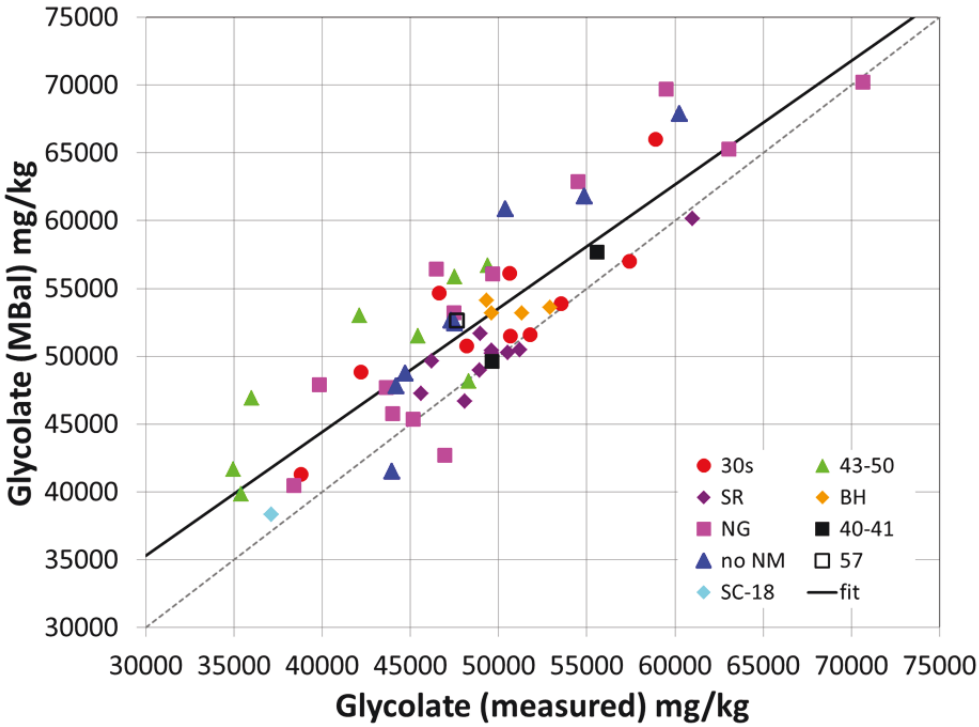


Figure 1. Material Balance versus Measured Glycolate

2.3 Input Data Variability

Variability plots of the input data are shown in Figures 2 and 3. The range of each data set is shown individually in Figure 2 and all of the series combined that were actually used in the final models (SR+BH, 43-50,57, SB9-NG, and also SC-18) are given in Figure 3. These plots show the variability of the data by data group. The boxes contain 75% of the data points; data points beyond the 'whiskers' outside the boxes might have relatively high leverage. The 43-50 and SB9-NG series examined a wide range of AS values, including less than 100%. The other runs were all performed at greater than 100% AS. The PRA values of the SB9-NG and SR+BH runs covered a similar range of ~52-60% with the SB9-NG series including some values up to 65%. The 30s series were all done at relatively high PRA because the glycolate analysis method at this time produced values that were too low. The low glycolate values resulted in addition of more glycolic acid than actually required. The glasses made from these runs were all very reducing, confirming too much glycolate was present.

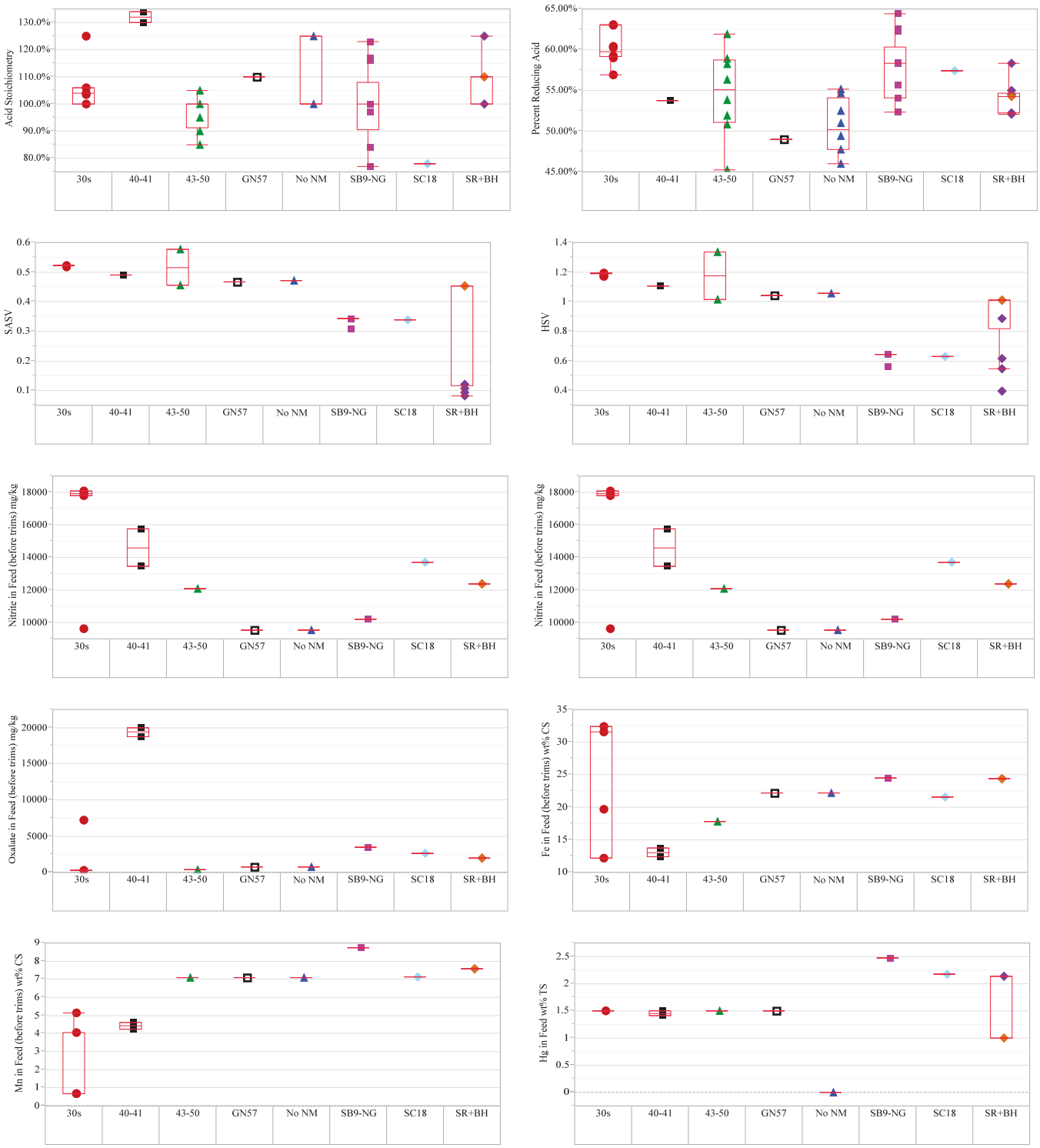
In addition to studying a wide range of AS values, the 43-50 series had a wide range of PRA values. The No NM series used less reducing acid to counteract the fact that the products were found to contain significantly more formate. The SASV and HSV values are discussed in Section 2.4. The nitrite and nitrate concentrations for SB9-NG and SR+BH approximated actual sludge batch compositions, whereas the 43-50 series were somewhat more 'generic' compositions. The 40-41 series simulated less-washed sludges, thus both nitrite and nitrate were higher. Nitrite and nitrate in the 30s series were exceptionally high except for one run; these simulants were not designed to mimic any particular sludge batch or extent of washing. The No NM series and Run 57 used relatively low nitrite to nitrate ratio.

The oxalate concentrations for most tests were low, except for the less-washed 40-41 series with very high oxalate. Iron values for most of the runs were in the typical 22-25 wt % CS range. Series 43-50 were lower, while series 40-41 were very low because the simulant was less-washed. The 30s series varied Fe and Mn artificially over a wide range. Manganese for all but the 30s series and 40-41 series was in the range from about 7-8.8%. Mercury was at 1.5 wt % for four of the series, while it was at about 2.1 wt %

for the SR series approximating SB8 and at 2.5 wt % in SB9-NG. The BH series, done with the same simulant as the SR series, was performed with the lower Hg concentration of 1.0 wt % to reduce the steam stripping duration.

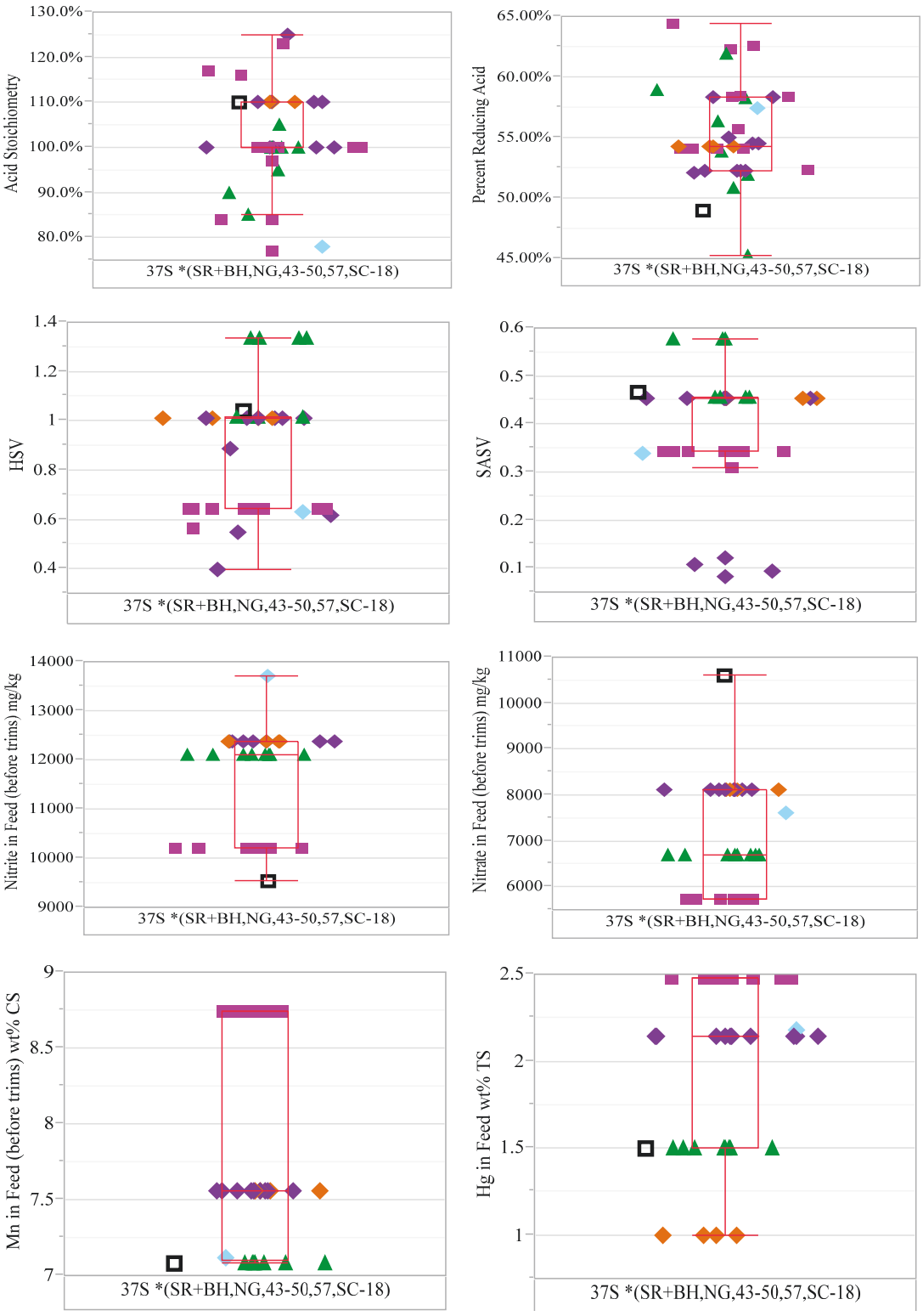
The combined plots in Figure 3 are useful to see what data points are significant outliers and would have greater leverage on the regressions. (The location of the data points on the x-axis is arbitrary; they are just distributed by JMP for added visual clarity.) Distant points can have more leverage than a grouping of points over a small region. For AS, most of the data points lay between 95-120%, with 4 SB9-NG series points further away since this test was performed to test the extremes of AS, PRA, and REDOX. The 43-50 series also tested lower values of AS; two runs had AS values of 90% or less. These points and the SR point at 125% will have greater leverage than the many points between 95-120%. Note that the SC-18 run was performed at the low end of the AS range. PRA values were mostly all between 50-60%, but several 43-50 and SB9-NG values were further away. Run 57 was also at the lower end of PRA.

The SC-18 nitrite concentration was significantly higher than any of the nitrite concentrations used in the modeling. The nitrite concentrations for the No NM series and Run 57 were at the lower end of the values, but were not significantly lower. The nitrate values for the No NM series and Run 57 were significantly higher than all of the other runs, which gives these nitrate values greater leverage in regression.



(see Table 1 for color key)

Figure 2. Input Variables Data Variability Versus Data Set



(see Table 1 for color key)

Figure 3. Combined Input Variables Data Variability

2.4 Headspace Variables

The variables SASV and HSV were chosen to potentially represent the region of the vapor headspace in the SRAT where internal refluxing could occur. Internal refluxing is the condensation of evaporated water as it cools in the headspace vapor or on the surfaces of the vessel headspace (walls, top head and components). It has been postulated that chemical reactions of offgas species occur in the headspace or on the surfaces of the headspace. The reactions most suspected of occurring are reactions of NO_x species with water to form nitrous and nitric acids that then condense and return to the SRAT vessel without the NO_x species actually ever leaving the vessel.

During acid addition nitric oxide (NO) is the most likely offgas species formed directly from the decomposition of nitrite in the simulant. Some direct formation of NO_2 may also occur. The following reactions describe this chemistry.

$$\text{SRAT liquid: } 3\,HNO_2 \rightarrow HNO_3 + 2\,NO + H_2O \quad (1)$$

$$\text{Vapor space: } NO + \tfrac{1}{2}O_2 \rightarrow NO_2 \quad (2)$$

$$\text{Vapor space or vapor space surfaces: } 2NO_2 + H_2O \rightleftarrows HNO_2 + HNO_3 \quad (3)$$

$$\text{Vapor space surface: } 3\,HNO_2 \rightarrow HNO_3 + 2\,NO + H_2O \quad (4)$$

The vapor space formation of nitrous and nitric acids is actually much more complex than indicated by Reaction 3, but this equation summarizes the overall reactions occurring.

Reactions 3 and 4 also occur in the SRAT condenser, and during reflux, nitric acid is returned to the SRAT vessel. The formation and reflux of HNO_3 in the SRAT condenser and in the headspace are indistinguishable. SRAT condenser condensation composition measurements versus time would be needed to determine the amount of HNO_3 formed by internal refluxing. It has been assumed that the amount of scrubbing of NO_x in the SRAT condenser in the experimental tests was the same relative to the simulant volumes, so that there was no effect of scale on the relative amount of HNO_3 returned to the SRAT.

The reason for proposing an effect of the vapor headspace on the reactions was that there appeared to be differences in the nitrite to nitrate ratio in the SR series that correlated with the vessel size (4-L, 22-L, and 220-L). The actual volume of the vessel did not make sense as a variable affecting nitrite to nitrate conversion, so the variables SASV and HSV were proposed. HSV was proposed because it is the ratio of vapor headspace available for Reactions 2-4 to the volume of simulant. The volume of simulant was used because it is the source term for the offgases, i.e., the amount of offgas species is dependent on the amount of simulant used. The SASV variable is the ratio of the headspace total surface area to the simulant volume and thus incorporates Reactions 3-4 on the surfaces. In reality, the 'variable' that might be best for correlating the data could be some combination of these two variables. However, due to how scattered the data are, such distinctions were not possible.

The headspace surface areas and volumes for all of the series and for DWPF are shown in Table 8. A summary of these calculations is given in Appendix A; the detailed calculations are archived in the ELN. The HSV and SASV ratios are plotted in Figure 4. Although these variables are smaller for the larger-vessel tests, this relationship is not necessarily a constant for a given size. For example, if the 4-L vessels used were filled significantly more, the amount of headspace could decrease to values like those for the larger vessels. The reason runs are not done this way is that overfilling results in much less vertical headspace for foam to dissipate and is thus not desirable from an operational standpoint.

Table 8. Headspace Surface Area and Volume Data

Vessel Size	Runs	Simulant Volume (mL)	Headspace Volume (mL)	Headspace Surface Area (cm^2)	Headspace Surface Area to Sludge Volume (SASV) (cm^{-1})	Headspace Volume to Sludge Volume (HSV)
DWPF	-	31,600,000	13,000,000	284,000	0.00901	0.411
220-L	GN78	115,000	102,000	12,400	0.108	0.888
220-L	GN79	134,000	82,700	11,100	0.0825	0.617
22-L	GN76	17,600	6,960	1,660	0.0940	0.395
22-L	GN77	15,900	8,690	1,900	0.120	0.547
4-L	GN70-75	3,040	3,070	1,380	0.454	1.01
4-L	GN80-83 (BH)	3,039	3,069	1,379	0.454	1.01
4-L	SB9-NG-51-61	3,068	1,976	1,052	0.343	0.644
4-L	SB9-NG-62	3,232	1,811	1,811	0.310	0.560
		Average Values				
4-L	GN34-38	2,790	3,318	1,454	0.521	1.19
4-L	GN40-41	2,900	3,207	1,421	0.490	1.11
4-L	GN43-46	2,614	3,493	1,507	0.576	1.33
4-L	GN47-5 0	3,030	3,077	1,382	0.456	1.02
4-L	GN51-59	2,974	3,133	1,399	0.470	1.05

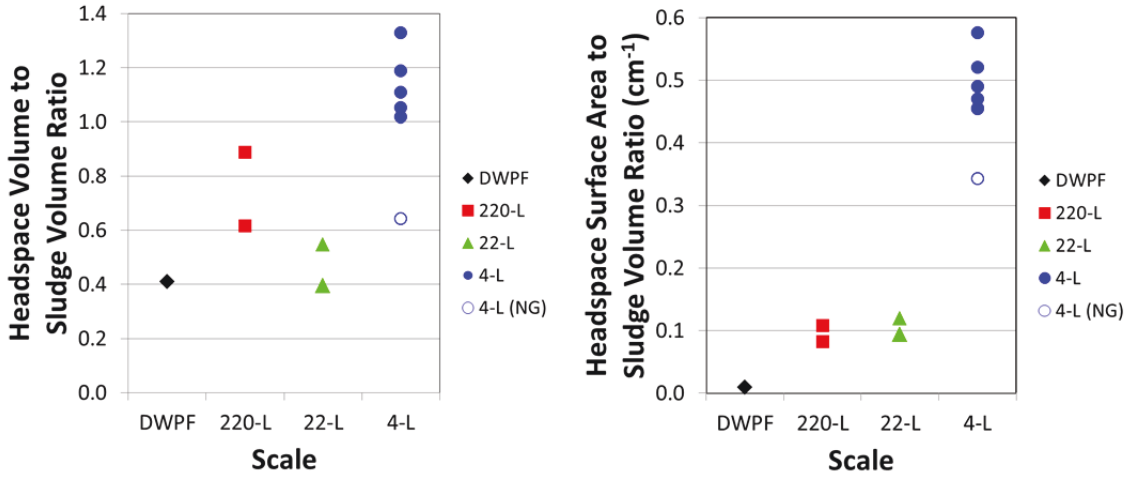


Figure 4. HSV and SASV for DWPF and Simulant Runs

SASV are significantly higher for the 4-L runs, meaning the headspace in these runs is a significantly larger portion of the vessel than in the larger vessels. The SB9-NG series, using a new flat metal top head, had the lowest SASV values for 4-L vessels. The HSV values for these runs are the only ones with a magnitude similar to the larger scale vessels. The 4-L vessels are relatively tall and slender whereas the larger vessels have a height that is closer to the vessel diameter. The original 4-L vessels had a domed top head with a volume of almost 1,000 mL, whereas the new top head is flat and adds no volume. The new top heads make the 4-L vessels very close to prototypic with respect to HSV and a bit closer with respect to SASV.

The variable HSV is similar for DWPF and the 22-L tests, while the 220-L values are higher but still only about half the 4-L values. If any conversions are truly proportional to HSV, then DWPF behavior would be expected to be similar to the 22-L runs.

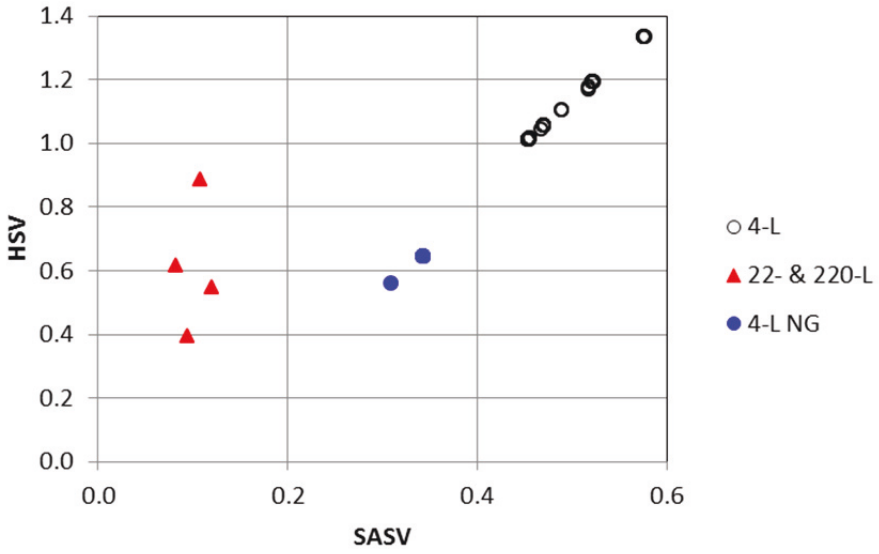


Figure 5. HSV versus SASV

The variable SASV is similar for the 22-L and 220-L runs and is about 1/5 of the values for the 4-L runs except for the SB9-NG runs. Correlation to SASV would imply that the 22-L and 220-L vessels behaved similarly. However, the DWPF value is about 1/10 the 22- and 220-L values. This difference is significantly greater than the HSV values and would predict very different behavior in DWPF relative to the simulant test equipment. The reason the DWPF SASV value is so small is that SASV will always be smaller in a similarly configured larger vessel. SASV decreases by the reciprocal of the radius, whereas HSV is only dependent on the detailed dimensions and is not necessarily a function of the volume.

Since both of these variables are empirically derived to possibly capture the effect of different headspace geometries, there are no definitive reasons to pick one over the other.

2.5 Nitrite to Nitrate Conversion

The nitrite to nitrate conversion was calculated from the initial amount of nitrite and nitrate in the simulant and the amount of nitrate in the final product (SRAT or SME). Define nitrite to nitrate conversion (N_C) as:

$$N_C = \frac{n^P_{NO_3^-} - n^F_{NO_3^-} - n^{HNO_3}_{NO_3^-}}{n^F_{NO_2^-}} \qquad 5$$

where $n^P_{NO_3^-}$ = moles of nitrate in product

$n^F_{NO_3^-}$ = moles of nitrate in feed

$n^{HNO_3}_{NO_3^-}$ = moles of nitric acid added

$n^F_{NO_2^-}$ = moles of nitrite in feed

The following equation is then used to determine the amount of nitrate in the product:

$$n^P_{NO_3^-} = N_C n^F_{NO_2^-} + n^F_{NO_3^-} + n^{HNO_3}_{NO_3^-} \qquad 6$$

In the Part 1 report,[1] the nitrite to nitrate conversion was expressed in terms of an alternate formulation called N_R. For this Part 2 report, N_R will not be used, but is described below to allow comparisons to the Part 1 report. The fraction of the total original nitrite + nitrate + HNO_3 added that becomes nitrate N_R is:

$$N_R = \frac{n^{P}_{NO_3^-}}{n^{F}_{NO_3^-} + n^{HNO_3}_{NO_3^-} + n^{F}_{NO_2^-}} \quad 7$$

To calculate N_R from N_C:

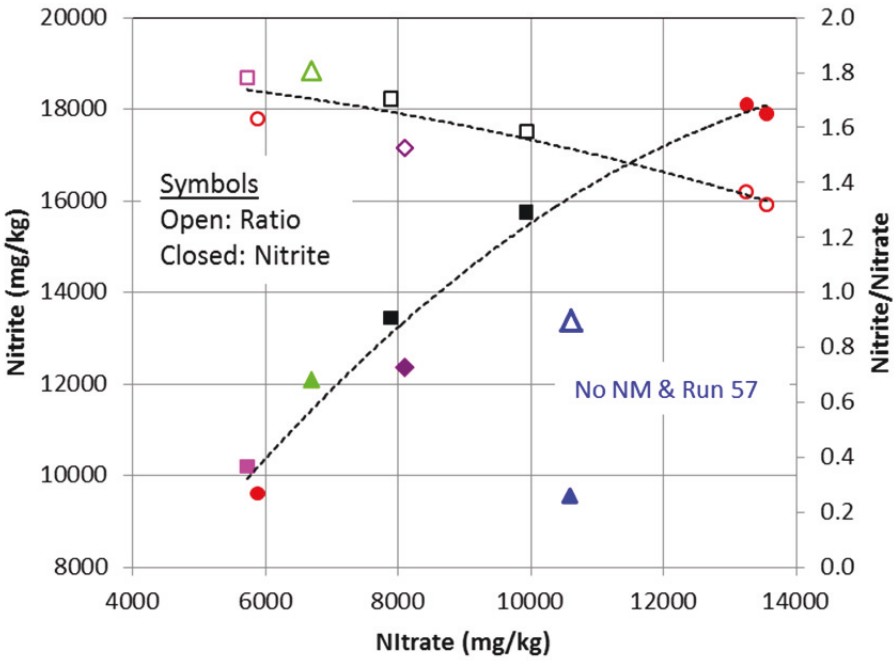
$$N_R = \frac{N_C n^{F}_{NO_2^-} + n^{F}_{NO_3^-} + n^{HNO_3}_{NO_3^-}}{n^{F}_{NO_3^-} + n^{HNO_3}_{NO_3^-} + n^{F}_{NO_2^-}} \quad 8$$

2.6 Correlation of Experimental Variables

Correlations between nitrite and nitrate and between Mn and Fe were found with the exception of certain data series. The correlation of nitrite and nitrate is shown in Figure 6. For all data series except the No NM series and Run 57, nitrite and nitrate were extremely correlated, and the nitrite to nitrate ratio varied from about 1.32 to 1.83. This correlation is due to the tank farm maintaining an excess of nitrite relative to nitrate for corrosion control. Because of this correlation, it is expected that any dependence of data on nitrite would have a similar dependence on nitrate. In the No NM series and Run 57 this ratio was 0.90. Only the data from the No NM series and Run 57 would contribute any information to discerning a difference in dependence on nitrite versus nitrate. For the most part, a robust model could contain either nitrite or nitrate, but not both.

The correlation of Mn and Fe concentrations is shown in Figure 7. The 30s series (Mn:Fe matrix study) demonstrate the wide range of compositions tested. Only the composition at about 11 wt % Fe and 3 wt % Mn is an expected composition in a waste tank; the others were just extreme cases used to determine the effects of Mn and Fe separately from each other. The remaining data from all other series are reasonably well correlated, so without the 30s series, models depending on Mn or Fe should be fit about equally well with either variable.

Based on the observed chemistry, a dependence on Mn is expected to be more likely than one on Fe because MnO_2 (Mn^{4+}) is known to be significantly reduced to Mn^{2+} whereas Fe^{3+} appears to be reduced much less to Fe^{2+}. (It has not yet been determined if solid Fe^{3+} species are being converted to solid Fe^{2+} species in the NG flowsheet.)



(Symbol shapes and colors correspond to key in Table 1, except Run 57, which is shown The same as No NM because it was the same composition.)

Figure 6. Correlation of Nitrite and Nitrate

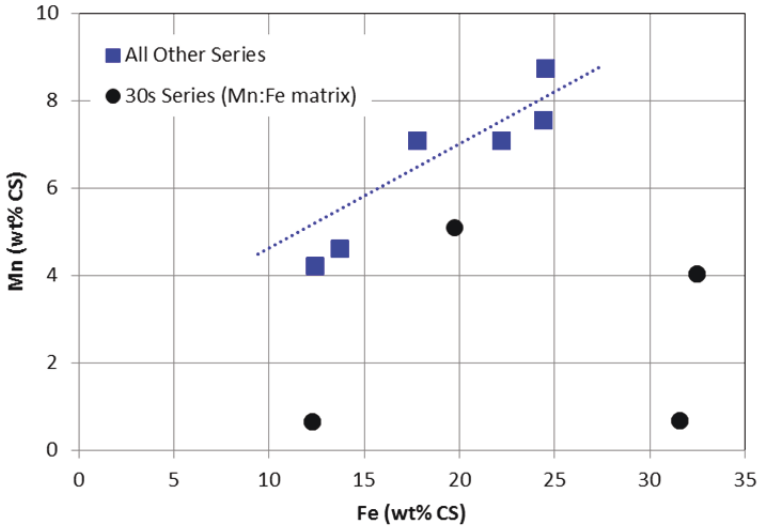


Figure 7. Correlation of Mn and Fe

Further correlation of variables can be found when only one or two data series are combined because most data series have only one value for each variable. With just two series, any two values define a line, a statistical dependence can be assigned to any variable that is linear. For example, if nitrite values are 10,000 and 15,000 mg/kg and Mn values are 7.5 and 7.0%, respectively, both nitrite and Mn could be equally statistically significant since nitrite and Mn are related linearly by nitrite = $-1x10^{-6}$ * Mn(%) + 85000.

The correlations of input variables in the SR+BH, SB9-NG, and 43-50,57 series are shown in Table 9. Pairs of data series are shown in the first five groupings in these two tables, while two groups of three are shown in the last two. The groupings are lettered A-G for easier referencing. AS and AS*PRA are somewhat correlated as would be expected. For grouping A, there is total correlation between nitrate, oxalate, Fe, and Mn, meaning it is not possible to distinguish between effects of these variables by only statistic methods. Grouping C has similar correlation but in addition Hg is totally correlated with these variables, and SASV and HSV are unexpectedly correlated with all variables except AS, PRA, and AS*PRA. For this grouping, SASV and HSV are also totally correlated. In grouping B there is less correlation because there are three distinct compositions of simulant, but there are still a large number of variables that are correlated. The correlation in grouping E is very similar to A. There is much less correlation in grouping D.

Table 9. Correlations Between Variables in Groups of Series

(A) **Data Sets: SR+BH SB9-NG**

	AS	PRA	ASxPRA	SASV	HSV	Nitrite	Nitrate	Oxalate	Fe	Mn	Hg
AS	1	-0.23	0.85	0.13	0.34	0.35	0.35	-0.35	-0.35	-0.35	-0.31
	PRA	1	0.32	-0.05	-0.40	-0.49	-0.49	0.49	0.49	0.49	0.32
		ASxPRA	1	0.10	0.11	0.06	0.06	-0.06	-0.06	-0.06	-0.12
			SASV	1	0.70	0.06	0.06	-0.06	-0.06	-0.06	-0.33
				HSV	1	0.66	0.66	-0.66	-0.66	-0.66	-0.64
					Nitrite	1	1.00	-1.00	-1.00	-1.00	-0.67
						Nitrate	1	-1.00	-1.00	-1.00	-0.67
							Oxalate	1	1.00	1.00	0.67
								Fe	1	1.00	0.67
									Mn	1	0.67
										Hg	1

Key:

> 0.99
0.95<X≤0.99
0.90<X≤0.95
0.8≤X≤0.90

(B) **Data Sets: SB9-NG 43-50,57**

	AS	PRA	ASxPRA	SASV	HSV	Nitrite	Nitrate	Oxalate	Fe	Mn	Hg
AS	1	-0.10	0.78	-0.15	-0.14	-0.19	0.14	0.08	0.13	0.07	0.07
	PRA	1	0.55	-0.25	-0.26	-0.17	-0.43	0.35	0.28	0.37	0.37
		ASxPRA	1	-0.28	-0.29	-0.26	-0.17	0.29	0.29	0.29	0.29
			SASV	1	1.00	0.82	0.52	-0.91	-0.90	-0.91	-0.91
				HSV	1	0.83	0.54	-0.93	-0.92	-0.93	-0.93
					Nitrite	1	0.15	-0.87	-0.96	-0.84	-0.84
						Nitrate	1	-0.62	-0.43	-0.66	-0.66
							Oxalate	1	0.97	1.00	1.00
								Fe	1	0.96	0.96
									Mn	1	1.00
										Hg	1

(C) **Data Sets: SB9-NG 43-50**

	AS	PRA	ASxPRA	SASV	HSV	Nitrite	Nitrate	Oxalate	Fe	Mn	Hg
AS	1	-0.02	0.81	-0.19	-0.18	-0.13	-0.13	0.13	0.13	0.13	0.13
	PRA	1	0.56	-0.22	-0.23	-0.31	-0.31	0.31	0.31	0.31	0.31
		ASxPRA	1	-0.28	-0.28	-0.29	-0.29	0.29	0.29	0.29	0.29
			SASV	1	1.00	0.91	0.91	-0.91	-0.91	-0.91	-0.91
				HSV	1	0.93	0.93	-0.93	-0.93	-0.93	-0.93
					Nitrite	1	1.00	-1.00	-1.00	-1.00	-1.00
						Nitrate	1	-1.00	-1.00	-1.00	-1.00
							Oxalate	1	1.00	1.00	1.00
								Fe	1	1.00	1.00
									Mn	1	1.00
										Hg	1

Table 9 Continued Correlations Between Variables in Groups of Series

(D) Data Sets: SR+BH 43-50,57

	AS	PRA	ASxPRA	SASV	HSV	Nitrite	Nitrate	Oxalate	Fe	Mn	Hg
AS	1	-0.27	0.72	-0.22	-0.30	-0.02	0.55	0.57	0.61	0.55	0.05
	PRA	1	0.47	0.03	0.02	0.29	-0.26	-0.01	-0.07	0.02	0.00
		ASxPRA	1	-0.18	-0.26	0.21	0.31	0.52	0.51	0.52	0.05
			SASV	1	0.91	-0.18	-0.32	-0.51	-0.51	-0.51	-0.49
				HSV	1	-0.16	-0.40	-0.58	-0.58	-0.57	-0.43
					Nitrite	1	-0.50	0.38	0.21	0.47	0.17
						Nitrate	1	0.61	0.74	0.53	0.19
							Oxalate	1	0.98	1.00	0.35
								Fe	1	0.96	0.34
									Mn	1	0.36
										Hg	1

(E) Data Sets: SR+BH 43-50

	AS	PRA	ASxPRA	SASV	HSV	Nitrite	Nitrate	Oxalate	Fe	Mn	Hg
AS	1	-0.23	0.75	-0.23	-0.31	0.62	0.62	0.62	0.62	0.62	0.07
	PRA	1	0.47	0.06	0.03	-0.07	-0.07	-0.07	-0.07	-0.07	-0.03
		ASxPRA	1	-0.17	-0.26	0.52	0.52	0.52	0.52	0.52	0.04
			SASV	1	0.91	-0.51	-0.51	-0.51	-0.51	-0.51	-0.49
				HSV	1	-0.58	-0.58	-0.58	-0.58	-0.58	-0.43
					Nitrite	1	1.00	1.00	1.00	1.00	0.35
						Nitrate	1	1.00	1.00	1.00	0.35
							Oxalate	1	1.00	1.00	0.35
								Fe	1	1.00	0.35
									Mn	1	0.35
										Hg	1

(F) Data Sets: SR+BH SB9-NG 43-50,57

	AS	PRA	ASxPRA	SASV	HSV	Nitrite	Nitrate	Oxalate	Fe	Mn	Hg
AS	1	-0.20	0.79	-0.08	0.00	0.16	0.36	0.03	0.27	-0.09	-0.12
	PRA	1	0.44	-0.09	-0.27	-0.28	-0.44	0.35	0.13	0.40	0.31
		ASxPRA	1	-0.13	-0.18	-0.03	0.04	0.25	0.32	0.18	0.09
			SASV	1	0.83	0.18	0.04	-0.50	-0.56	-0.41	-0.53
				HSV	1	0.57	0.38	-0.81	-0.67	-0.78	-0.72
					Nitrite	1	0.59	-0.68	-0.31	-0.81	-0.62
						Nitrate	1	-0.49	0.06	-0.68	-0.52
							Oxalate	1	0.83	0.96	0.76
								Fe	1	0.65	0.53
									Mn	1	0.78
										Hg	1

(G) Data Sets: SR+BH SB9-NG 43-50

	AS	PRA	ASxPRA	SASV	HSV	Nitrite	Nitrate	Oxalate	Fe	Mn	Hg
AS	1	-0.18	0.81	-0.09	-0.02	0.22	0.35	0.05	0.28	-0.06	-0.10
	PRA	1	0.44	-0.07	-0.25	-0.40	-0.36	0.31	0.13	0.37	0.28
		ASxPRA	1	-0.12	-0.17	-0.05	0.09	0.24	0.32	0.17	0.08
			SASV	1	0.82	0.22	-0.02	-0.49	-0.56	-0.40	-0.52
				HSV	1	0.64	0.38	-0.80	-0.68	-0.77	-0.72
					Nitrite	1	0.91	-0.80	-0.34	-0.93	-0.71
						Nitrate	1	-0.46	0.09	-0.69	-0.51
							Oxalate	1	0.84	0.96	0.75
								Fe	1	0.66	0.53
									Mn	1	0.77
										Hg	1

Groupings F and G show all three data series combined, with F including Run 57 and G not. The only very high correlation is between oxalate and Hg, with less between oxalate with HSV, nitrite, and Mn.

2.7 Quality Assurance

Requirements for performing reviews of technical reports and the extent of review are established in manual E7 2.60. SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011, Rev. 2.

These reviews were conducted by J.D. Newell.

3.0 Results and Discussion

3.1 General Formulation of Models

The models used for nitrite conversion to nitrate expressed either as N_C or N_R, glycolate destruction (G_D), glycolate conversion to formate + CO_2 (G_F), and glycolate conversion to oxalate (G_{Ox}) are empirical correlations of these data versus the variables in Table 4. All of the data used for the models are the MBal data, unless otherwise noted. The models considered are all linear in the variables except for the addition of the cross product (AS*PRA). The AS*PRA represents the portion of the AS that is the reducing acid glycolic acid; it could be called the 'Reducing Acid Stoichiometry' with the remainder being the nitric acid that causes only acid base reactions and not REDOX reactions such as the reduction of Hg or Mn. In Part 1[1] of this study, numerous cross products and squares (e.g., nitrite*AS, AS^2) were tried. These higher order terms were often found to improve the fit of the data but added too many predictive terms to the models to be reliable for future predictions. Therefore, higher order terms other than AS*PRA were not used.

The general strategy for modeling the data were to first fit each individual data set to the best model from stepwise regression in JMP. Commonalities between the models were noted that should be in models of combined data series. The data series were combined in groups of 2, 3, and more. Table 10 shows the groupings. Most of the data series had no differences in composition so nitrite, nitrate, Mn, etc. cannot be used to fit the data. For most but not all series, HSV and SASV did not vary significantly. All series had variations in AS and PRA. (For brevity, nI will be used to abbreviate nitrite, nA will be used for nitrate, and Ox for oxalate.) For example, the SR data can only be fit versus AS, PRA, SASV and HSV since no other variables vary. The addition of BH to make SR+BH brings in Hg as a possible variable to distinguish between the data series. The addition of more variables is needed to distinguish between the individual data series that are fit with less variables.

Table 10. Data Groupings for Regression

Number of Data Series in Grouping	Data Set Groups	Number of Data Points	Additional Variables (+AS,PRA)
1	30s	10	Mn, Fe nI, nA (1 run)
	43-50	8	SASV, HSV
	No NM (51-56,58-59)	8	None
	SR	10	SASV, HSV
	SB9-NG	13	None
	43-50,57*	9	SASV, HSV, nI, nA, Mn, Fe, Ox
	SR+BH**	14	SASV, HSV, Hg
2	43-50,57 + SB9-NG	22	All Variables
	43-50,57 + SR+BH	23	
	SR+BH + SB9-NG	27	
3	43-50,57 + SB9-NG + SR+BH	36	
4	43-50,57 + SB9-NG + SR+BH + 40-41	38	
	43-50,57 + SB9-NG + SR+BH + No NM	44	
5	43-50,57 + SB9-NG + SR+BH + No NM + 40-41	46	
6	43-50,57 + SB9-NG + SR+BH + No NM + 40-41 + 30s	56	

* Because Run 57 was a lone run (same composition as No NM, but with Hg and NM), it was often combined with 43-50.

** Because the SR and BH tests used the same simulant (except for Hg concentration), these runs were treated together.

Stepwise regression relying on JMP's P-value Threshold stopping rule was used to select a subset of the candidate effects to develop a regression model of potential interest for various groups of the experimental data. The P-value Threshold, a part of JMP's Stepwise regression routine, uses P-values (significance levels) to enter and remove effects from the model. The Probability to Enter is the maximum P-value that an effect must have to be entered into the model during a forward step of the stepwise regression routine. This is the default value used by JMP and as such it was the value utilized in this study. In general, the P-value for a term in a fitted model is a measure of the statistical significance: a small value (typically, less than or equal to 0.05) indicates a statistically significant term. JMP's default entrance P-value of 0.25 was used as the criterion for a candidate term to be considered during a forward step (JMP's default direction) of the fitting algorithm and subsequently to be entered into the model. Upon the completion of the stepwise routine, the "Make Model" feature available as part of the platform was used to fit the resulting model, which contained the active terms determined by JMP's stepwise routine. Although statistical significance is generally assumed to mean a P-value of less than 0.05, higher P-values were accepted in this study because the empirical models did not have high R^2 values.

3.2 A Note About Empirical Data Fitting and R^2 Values

Empirical data fitting potentially suffers from the problem of incorrectly assigning causation to correlation. As will be shown, the data considered can be fit by a number of complex models that tend to over-specify the regression and result in correlated parameters (coefficients). This behavior can occur when additional variables are added that do not truly improve the fit of the data, but do increase the R^2 value. Complex combinations of variables can often be found to fit almost any data set. In the cases described below, for some data series many combinations of three variables or four variables are found to fit the data equally well, bringing into question which ones are really needed. All data fitting was done to linear models except for the addition of the variable AS*PRA, which might be considered the reducing acid stoichiometry. No higher order (e.g., AS^2) or other cross product terms were used.

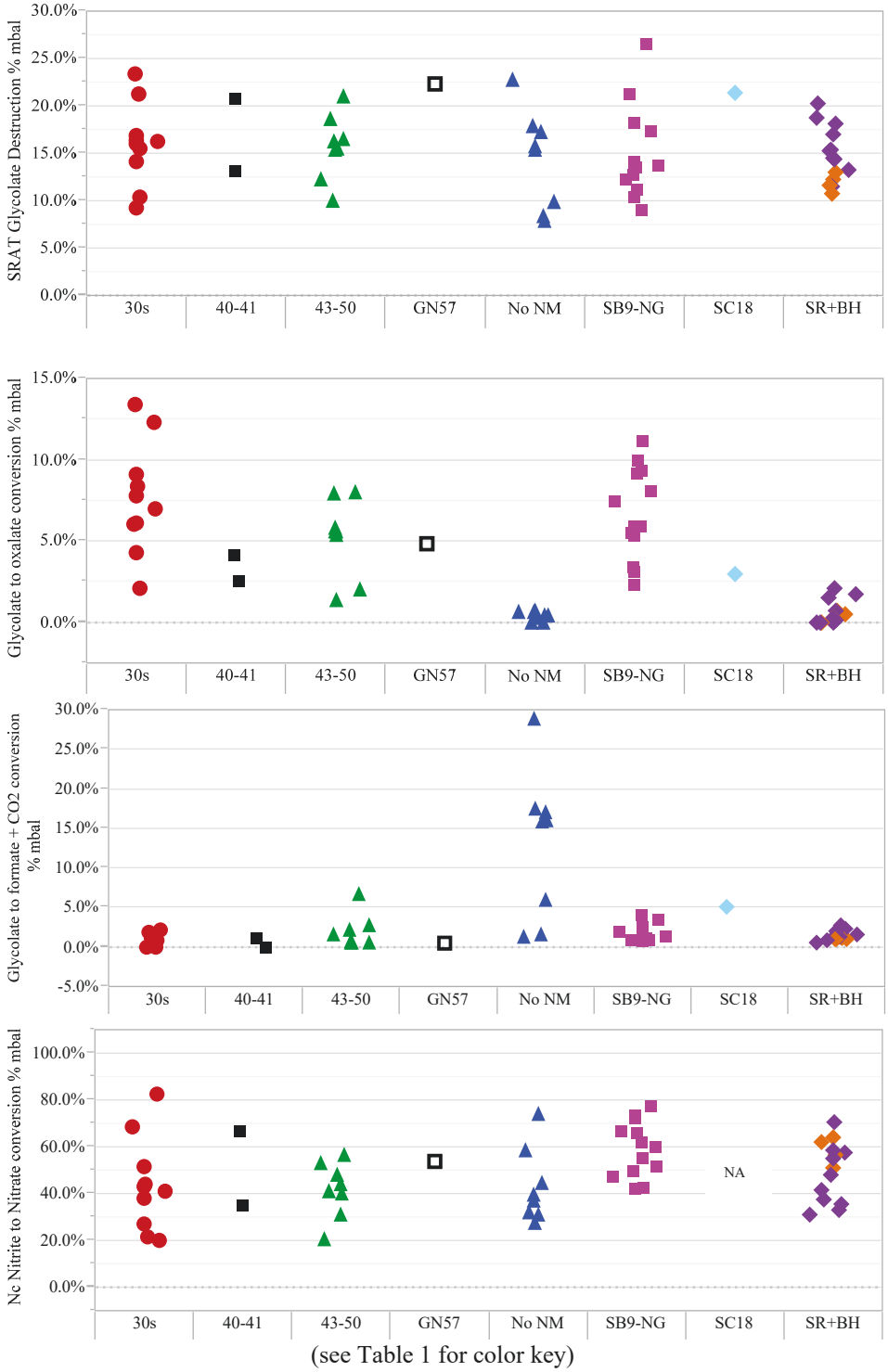
One of the difficulties that was found was that different series of data were fit by different variables. If one set of data varied (in addition to AS and PRA) only HSV, then HSV is the only other variable that can be used to regress the data. For another set, Hg could be varied. If both are found to be significant for their data set, then one would expect them to also be significant when the two data series are combined. However, the effect seen for one data set was often found to be less significant for combined series due to 'dilution' of the effect. For two series that are described by the same variable, such as AS, but the values of the estimated parameters differ between the series (same slope, different intercept), some other variable is needed to describe this difference. In the modeling done, this was usually when one of the composition variables nitrite, nitrate, Mn, Fe, or one of the noble metals was found to be significant. At this point it is difficult to decide whether to use the simpler less variable model or keep a more complex one that retains variables that were important in the fit of the individual series or groups of series. Another problem encountered when fitting a group of two or three series is the addition of two variables by the regression routine. Neither by themselves is sufficient to improve the predictions, but the linear combination of the two becomes significant. In cases like this, it is hard to attach any real significance to this addition of two variables.

In this report, R^2 and adjusted R^2 values are used to determine the adequacy of models. The R^2 value supposes that every independent variable in the model explains the variation in the dependent variable. It gives the percentage of explained variation as if all independent variables in the model affect the dependent variable, whereas the adjusted R^2 gives the percentage of variation explained by only those independent variables that in reality affect the dependent variable. Every time you add a variable to a model, the R^2 increases, even if due to chance alone. It never decreases. Consequently, a model with more terms may appear to have a better fit simply because it has more terms. The adjusted R^2 compensates for the addition of variables and only increases if the new term enhances the model above what would be obtained by probability and decreases when a variable enhances the model less than what is predicted by chance. (This was proven with the glycolate destruction data by adding a variable that was only random numbers; the R^2 increased, but the adjusted R^2 decreased, indicating that the random numbers did not add any significance to the fit.)

3.3 Output (Response) Data Variability

The variabilities of the responses to be fit are shown in Figure 8. Most of the data tends to be spread out fairly evenly over an area. There is no a priori reason to believe that each data set would span a particular range since multiple variables were changed in the tests. However, it was generally found that in the fitting of the models, points that appear to be outliers in these plots tended to be difficult to fit. These more extreme points had more leverage in the model fitting than other points due to their values being significantly different from the bulk of the other values. For example, the glycolate destruction value for the SB9-NG series at about 27% had more leverage, and was found to not be fit well by any model. Similarly, the glycolate to formate conversion value for the No NM series at about 29% was a leverage point and was not fit well. In both of these cases, the significant difference from the bulk of the data suggests that these points may have truly been outliers and were probably inaccurate values.

The glycolate to oxalate conversions for the No NM and SR+BH series are significantly lower than the other runs. Most of the glycolate to formate conversions are less than 4%, but for the No NM series, the values range up to 29%. Notably, the conversion to formate in the SC-18 run is higher than most of the other data with noble metals and Hg present. The nitrite to nitrate conversion, expressed as N_C covers similar ranges.



(see Table 1 for color key)

Figure 8. Output Data Variability

3.4 Modeling of Glycolate Destruction

Glycolate destruction encompasses the formation of oxalate, formate, and CO_2. One mole of glycolic acid can form one mole of oxalic acid, as shown for example in Equation 9 for the reduction of MnO_2 (these equations are possible examples and do not imply that these reactions actually occur, or if they occur, to what extent). Equation 10 shows that glycolic acid can form one mole of formic acid and one mole of CO_2. The formic acid can react further to produce CO_2, so the overall reaction of glycolic acid to produce two moles of CO_2 would be as given by Equation 11.

$$2\ MnO_2 + C_2H_4O_3 + 4\ H^+ = 2\ Mn^{2+} + (COOH)_2 + 3\ H_2O \quad (9)$$

$$2\ MnO_2 + C_2H_4O_3 + 4\ H^+ = 2\ Mn^{2+} + HCO_2H + CO_2 + 3\ H_2O \quad (10)$$

$$3\ MnO_2 + C_2H_4O_3 + 6\ H^+ = 3\ Mn^{2+} + 2\ CO_2 + 5\ H_2O \quad (11)$$

The modeling of glycolate chemistry was broken down into the net destruction of glycolate (G_D) (to form oxalate, formate, or CO_2), the amount of glycolate that formed oxalate (G_{Ox}), and the amount of glycolate that formed formate + CO_2 (G_F). These quantities have been calculated as percentages:

G_{Ox} = oxalate formed / glycolate added

G_F = formate formed / glycolate added

Additionally, the amount of the glycolate destruction that forms oxalate or formate can be calculated:

Glycolate destruction to oxalate = G_{Ox}/G_D

Glycolate destruction to formate = G_F/G_D

The destruction of glycolate, conversion of glycolate to oxalate and formate, and conversion of nitrite to nitrate are summarized in Table 11 for both the Measured and MBal values. Within each data group, the runs are sorted ascendingly by AS. (Note that for nitrogen, when there was no material balance done, the measured values are shown in the material balance column since they were used as the material balance values. In most cases, the MBal glycolate destruction is less than the value from the measured data. The percentage of the glycolate destruction attributed to oxalate and formate + CO_2 in the products is shown in Table 12. The remaining percentage is the direct formation of CO_2. Within each data group, the runs have been arranged from lowest to highest AS. The SC-18 run data are shown on the graphs but these data were not included in fitting the models except for the conversion of glycolate to oxalate.

Except for the SR+BH series, the runs with noble metals and Hg had oxalate production values from 30-46%; the SR+BH series was anomalous with an average value of 4%, which was similar to the value for the No NM series. The formate attributed to the destruction of a mole of glycolate to create one mole of formate and CO_2 each ranged from 5-12% for the runs with noble metals. In the No NM series, this value for formate was 98% for the 100% AS runs, but was more similar (17%) to the runs with noble metals and Hg when the AS was 125%. For the runs with noble metals and Hg, the amount of CO_2 attributed to direct formation of two moles of CO_2 from one mole of glycolic acid ranged from 45-65% except for the SR+BH runs, where it was 87%. The No NM series at 100% AS had an average of ~0% direct CO_2 formation, but there was one -27% and one +33% value. The runs at 125% acid had considerably more direct CO_2 generation at 78%. Recall that the CO_2 generation was a rough estimate for the No NM series, so these material balance values are also very approximate. Regardless, it is obvious that considerably more formate and less oxalate are found in the products when noble metals are not present, and that higher AS results in less formate when there are no noble metals and Hg. When displayed as in Table 12, for the data with noble metals and Hg present, there does not appear to be any correlation between the amounts of oxalate generated with respect to the AS. However, when displayed as the percentage of the glycolate added that formed oxalate, oxalate does trend with AS.

Table 11. Nitrite to Nitrate Conversion and Glycolate Destruction and Conversion Values

	(Note data sorted within each series by AS)		N_C Nitrite to Nitrate Conversion		G_D Glycolate Destruction (Measured or CSBal, from IC Results)	G_D Glycolate Destruction (from Material Balance)	G_{Ox} Glycolate Conversion to Oxalate	G_F Glycolate Conversion to Formate and CO_2
Series	Run Number	Acid Stoich.	Material Bal.	Meas/ CSBal				
30s	GN35	100%	20.1%	NA	28.0%	23.4%	13.4%	2.1%
	GN37	100%	40.8%	NA	27.4%	16.0%	4.3%	0.0%
	GN37b	100%	21.7%	NA	17.6%	16.2%	6.0%	0.7%
	GN34b	104%	43.2%	NA	28.8%	16.4%	8.4%	1.2%
	GN34	104%	82.5%	NA	19.1%	9.3%	2.1%	0.0%
	GN34c	104%	38.1%	NA	25.0%	16.9%	9.1%	0.7%
	GN36	106%	68.7%	NA	25.2%	21.2%	12.3%	1.8%
	GN36b	106%	44.2%	NA	13.7%	14.1%	7.0%	0.8%
	GN36c	106%	27.2%	NA	16.0%	15.5%	7.8%	0.8%
	GN38	125%	51.4%	NA	9.7%	10.4%	6.1%	1.1%
40-41	GN41	130%	53.9%	NA	21.4%	13.1%	5.2%	0.7%
	GN40	134%	35.0%	NA	20.8%	20.8%	4.1%	1.1%
43-50	GN44	85%	44.0%	NA	32.6%	21.0%	8.0%	6.7%
	GN46	90%	48.2%	NA	20.6%	18.6%	7.9%	1.6%
	GN45	95%	40.0%	NA	25.0%	12.2%	2.0%	2.7%
	GN47	100%	31.2%	NA	27.7%	16.5%	5.6%	0.7%
	GN48	100%	20.5%	NA	28.4%	16.2%	5.8%	0.6%
	GN49	100%	40.9%	NA	36.2%	15.5%	5.4%	0.5%
	GN50	100%	56.4%	NA	30.6%	15.4%	5.6%	0.5%
	GN43	105%	52.9%	NA	29.2%	10.0%	1.4%	2.2%
No NM	GN51	100%	39.5%	NA	23.3%	15.4%	0.0%	15.9%
	GN52	100%	37.0%	NA	24.3%	15.8%	0.3%	16.0%
	GN54	100%	31.8%	NA	24.1%	17.3%	0.7%	17.0%
	GN55	100%	27.6%	NA	24.1%	17.9%	0.6%	17.4%
	GN56	100%	44.6%	NA	18.2%	22.8%	0.0%	28.8%
	GN53	125%	31.1%	NA	20.0%	9.9%	0.6%	6.0%
	GN58	125%	74.2%	NA	18.2%	7.9%	0.4%	1.3%
	GN59	125%	58.7%	NA	24.1%	8.4%	0.4%	1.5%
57	GN57	110%	53.9%	NA	21.4%	13.1%	5.2%	0.7%
SR	GN70	100%	30.9%	23.3%	17.6%	18.7%	1.5%	2.3%
	GN72	100%	41.3%	26.5%	19.9%	17.0%	0.3%	2.0%
	GN74	100%	35.6%	31.6%	20.5%	14.5%	0.0%	1.6%
	GN76	100%	32.8%	17.4%	20.4%	20.2%	2.1%	2.8%
	GN79	100%	37.6%	19.7%	15.7%	18.1%	1.7%	0.9%
	GN73	110%	58.7%	42.0%	17.9%	13.2%	0.0%	1.2%
	GN75	110%	55.0%	45.3%	14.9%	15.3%	0.7%	1.3%
	GN77	110%	57.4%	30.5%	15.9%	14.4%	0.0%	1.3%
	GN78	110%	48.1%	23.8%	16.5%	15.4%	0.1%	0.5%
BH	GN80	110%	56.5%	NA	15.4%	12.2%	0.0%	1.0%
	GN81	110%	63.9%	NA	12.8%	11.6%	0.3%	1.0%
	GN82	110%	51.1%	NA	18.8%	13.0%	0.5%	1.0%
	GN83	110%	62.2%	NA	18.7%	10.7%	0.7%	1.1%
SR	GN71	125%	70.5%	37.5%	10.3%	11.5%	0.2%	1.5%
SB9-NG	NG58	77%	47.0%	32.8%	19.2%	26.5%	11.1%	3.5%
	NG53	84%	42.2%	27.9%	27.1%	18.2%	8.1%	4.0%
	NG51	84%	42.3%	33.9%	21.7%	21.3%	9.2%	2.4%
	NG62	97%	59.9%	7.8%	26.8%	11.1%	9.3%	1.6%
	NG57	100%	51.5%	54.0%	13.2%	13.8%	5.9%	0.7%
	NG55	100%	49.6%	43.0%	25.1%	12.3%	5.5%	0.8%
	NG55A	100%	62.2%	98.5%	18.5%	14.0%	5.9%	1.2%
	NG56	100%	66.0%	53.9%	23.4%	13.5%	7.4%	0.9%
	NG60	100%	77.6%	52.5%	21.7%	14.1%	5.3%	1.1%
	NG61	100%	54.8%	47.2%	31.3%	17.4%	10.0%	0.8%
	NG52	116%	66.7%	66.2%	15.8%	12.8%	3.4%	1.5%
	NG54	117%	73.6%	62.1%	21.1%	9.0%	2.3%	0.9%
	NG59	123%	72.0%	119.2%	13.8%	10.4%	3.1%	1.9%
SC-18	SC-18	78%	NA		24.0%	21.4%	3.0%	5.0%

(see Table 1 for color key)

Table 12. Conversions of Glycolate to Oxalate and Formate

Series	Run Number	Acid Stoichiometry	Oxalate Generated / Glycolate Destroyed		Formate (+ CO_2) Generated / Glycolate Destroyed		Direct CO_2 Formation / Glycolate Destroyed	
30s	GN35	100%	57%	46%	9%	5%	34%	48%
	GN37	100%	27%		0%		73%	
	GN37b	100%	37%		4%		59%	
	GN34b	104%	51%		7%		42%	
	GN34	104%	22%		0%		78%	
	GN34c	104%	54%		4%		42%	
	GN36	106%	58%		8%		34%	
	GN36b	106%	49%		6%		45%	
	GN36c	106%	50%		5%		45%	
	GN38	125%	59%		11%		30%	
40-41	GN41	130%	19%	19%	0%	3%	81%	78%
	GN40	134%	19%		5%		75%	
43-50	GN44	85%	38%	31%	32%	12%	30%	56%
	GN46	90%	42%		8%		49%	
	GN45	95%	16%		22%		62%	
	GN47	100%	34%		4%		62%	
	GN48	100%	36%		4%		61%	
	GN49	100%	35%		3%		62%	
	GN50	100%	37%		3%		60%	
	GN43	105%	14%		22%		64%	
No NM	GN51	100%	0%	3%	103%	98%	-3%	-1%
	GN52	100%	2%		101%		-3%	
	GN54	100%	4%		99%		-3%	
	GN55	100%	4%		97%		0%	
	GN56	100%	0%		127%		-27%	
	GN53	125%	6%		61%		33%	
	GN58	125%	4%		17%	17%	79%	78%
	GN59	125%	5%		18%		76%	
	GN57	110%	40%	40%	5%	5%	55%	55%
SR	GN70	100%	8%	4%	12%	9%	80%	87%
	GN72	100%	2%		12%		87%	
	GN74	100%	0%		11%		89%	
	GN76	100%	10%		14%		76%	
	GN79	100%	9%		5%		86%	
	GN73	110%	0%		9%		91%	
	GN75	110%	5%		8%		87%	
	GN77	110%	0%		9%		91%	
	GN78	110%	1%		3%		96%	
	GN71	125%	2%		13%		85%	
BH	GN80	110%	0%		8%		92%	
	GN81	110%	2%		8%		89%	
	GN82	110%	4%		8%		88%	
	GN83	110%	7%		10%		83%	
SB9-NG	NG58	77%	42%	44%	13%	11%	45%	45%
	NG53	84%	44%		22%		34%	
	NG51	84%	43%		11%		45%	
	NG62	78%	83%		15%		2%	
	NG57	100%	43%		5%		52%	
	NG55	100%	45%		7%		48%	
	NG55A	100%	42%		9%		50%	
	NG56	100%	55%		7%		38%	
	NG60	100%	37%		8%		54%	
	NG61	100%	57%		4%		39%	
	NG52	116%	26%		12%		62%	
	NG54	117%	26%		10%		64%	
	NG59	123%	30%		18%		52%	
SC	SC-18	78%	15%	15%	21%	21%	64%	64%

(see Table 1 for color key)

As shown below, glycolate destruction was found to be a function of AS for all data series. This functionality is inherent in the definition of AS. An AS of 100% is a calculation of the minimum acid required to complete all the necessary reactions: neutralization of base and carbonate, destruction of nitrate, and reduction of Mn and Hg.

Because of the greater reducing power of glycolic acid compared to formic acid, the reactions for the reductions of Mn and Hg, and possibly the destruction of nitrite, are different for the NG flowsheet than for the NF flowsheet. For the NG flowsheet, it is known that the minimum acid calculated by the KMA formula is actually more than the minimum needed.

If it is assumed that the amount of glycolic acid that will be consumed for a given amount of a given simulant is a constant amount, then any excess will just decrease the percentage glycolate destruction by simple dilution. Figure 9 shows the percent glycolate destruction functionality versus AS for simple dilution. The values were calculated assuming that there was 15% glycolate destruction at 100% AS. Given the same absolute amount of glycolate destruction, the percent destruction was then calculated with less and greater than 100% AS. The functionality turns out to be G_D = (glycolate destruction % at 100% AS) / AS. The range of AS that has been studied is shown in Figure 9b. The hyperbolic function was fit to a line over the range from 70-135% AS. The graphs show that over this restricted range, the glycolate destruction is almost linear.

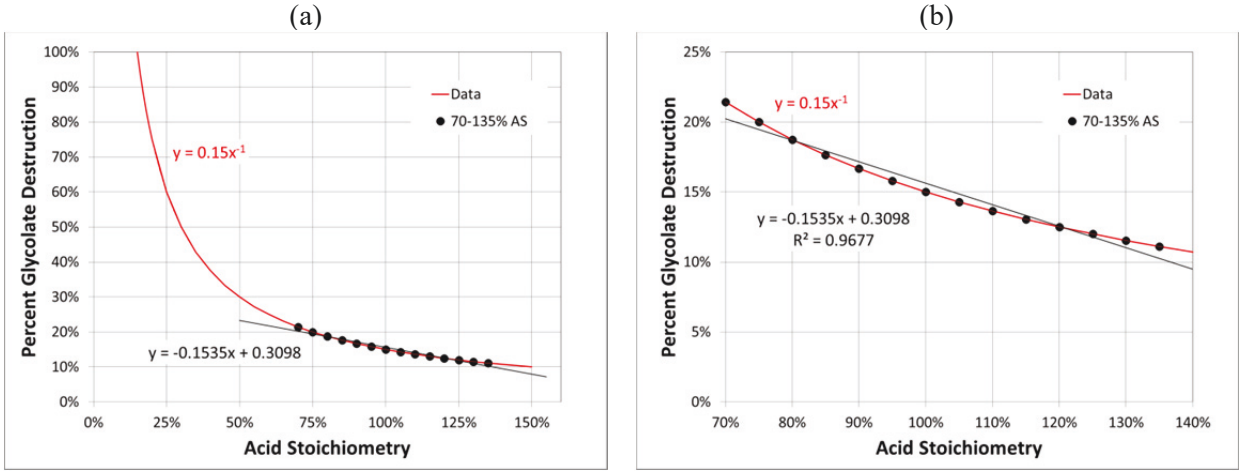


Figure 9. Glycolate Destruction Functionality versus Acid Stoichiometry

A comparison of glycolate destruction versus AS is shown for the Measured data and the MBal data in Figure 10. For the Measured data, there is no consistent relationship between glycolate destruction and AS, but for the MBal data, linear fits to each data set give approximately parallel lines (same slope) with different intercepts. The data are very scattered even within data series, but this graph shows that even without additional variables (beyond only AS) the dependence on AS is linear with similar slope. Differences between data series might be fit by variables that are different between the data series. The heavy black line shown in Figure 10 is the line from Figure 9 assuming 15% glycolate destruction at 100% AS. Note that this simple dilution functionality accounts for much of the trends in the data. The major difference is that the actual data have a steeper slope indicating that the absolute glycolate destruction is greater at lower AS and less at higher AS. The functionality of glycolate destruction on AS should probably be expressed versus 1/AS, but as Figure 9 (b) shows, the assumption of linearity deviates only a small amount from the hyperbolic function, and is well within the uncertainty of actual data.

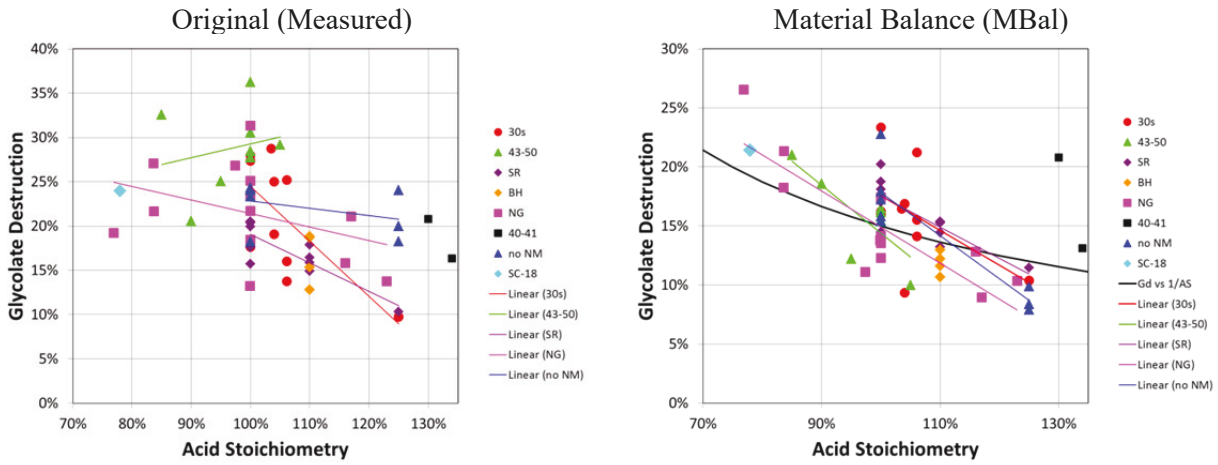


Figure 10. Glycolate Destruction versus Acid Stoichiometry

3.4.1 Fitting of Glycolate Destruction Models to Individual Data Series

Table 13 shows the color codes used for describing the significance of each variable in models. The significance measure is the probability that the coefficient on the variable is greater than the absolute value of the t-statistic. The t-statistic is the quantile of the Student's t probability distribution. In evaluating the effect of a parameter on a response, this distribution represents the likelihood, the P-value, of the regression outcome if the parameter has no effect on the response. If this P-value is 0.05 or smaller, there is a significant effect at the 5% significance level (i.e., the estimated effect is unlikely to be by chance alone). Generally, a probability of less than 0.05 is considered significant, but the choice of value is up to the user. In this work, larger P-values have been accepted because the R^2 values for the empirical regressions were low.

Table 13. Color Codes for Significance of Variables in Models

Parameter Significance Prob. > |t|	Color
Not included in model	
offered in stepwise but not significant	
P < 0.05	
0.05 < P <= 0.10	
0.10 < P <= 0.15	
0.15 < P <= 0.20	
P > 0.20	

The individual data series were fit using stepwise regression to determine what variables were statistically significant. The significant variables and R^2 values for each set are shown in Table 14; both the MBal and Measured data are shown. The fits of SR and SR+BH are also shown versus HSV rather than SASV, and SB9-NG is shown without SASV. The fits of the MBal data are shown in Figure 11.

Table 14. Glycolate Destruction Individual Data Series Stepwise Regression Significant Variables

Data Set	30s			43-50		SR			SR+BH			SB9-NG			No NM	
# Data Points	10			8		10			14			13			8	
Name	g30			g43		gS			gSB			gNG			gNo	
	(a)	(b)	(c)	(a)	(b)	(a)	(b)	(c)	(a)	(b)	(c)	(a)	(b)	(c)	(a)	(b)
	Mbal		meas	Mbal	meas	Mbal		meas	Mbal		meas	Mbal		meas	Mbal	meas
# Variables	1	2	2	2	1	3	3	1	4	4	1	2	1	1	1	none*
R^2	0.49	0.55	0.58	0.88	0.34	0.84	0.82	0.72	0.88	0.87	0.55	0.80	0.71	0.32	0.81	none
R2 adj	0.43	0.42	0.46	0.84	0.23	0.76	0.73	0.68	0.83	0.82	0.51	0.76	0.68	0.26	0.77	none
AS		0.05	0.07													
PRA		0.07			0.13	0.09	0.16		0.05	0.11						
AS*PRA																
SASV						0.16			0.12			0.06				
HSV							0.22			0.17						
nitrite																
nitrate																
oxalate																
Mn			0.19													
Hg																
Fe																

* regression was not significant ($R^2 = 0$)

For all data series, AS is significant. The AS*PRA is significant for the 30s series, but a fit without AS*PRA shows AS and PRA to be a bit less significant. Here is an example of an additional variable giving a higher R^2 but not adding a statistically significant improvement in the fit of the data as evidenced by the decrease in R^2-adjusted. The measured data fits are worse than the MBal data fits except for the 30s series; the No NM series measured data cannot be fit at all. Also note that there are less significant variables, and for example, the 43-50 series measured data are poorly fit by only PRA. The consistency of the significant variables for the MBal data is an indication that performing the material balance improved the data.

Additionally, the headspace variables HSV or SASV are somewhat significant for the SR, SR+BH, and SB9-NG data series. Note that for most data series, there were no significant differences in the values of nitrite, nitrate, oxalate, Mn, or Fe. For SR+BH, the same simulant was used except the Hg concentration was different for the BH series, and Hg was found to be significant.

The graphs of glycolate destruction measured versus predicted in Figure 11 show the data series that were not fit in a 'pastel' color while the series fit are in 'vivid' colors. The pastel data show the predicted values using each model for the data that was not fit. The graphs show that the model that fits a particular data set does not fit the other data series even though the important variables are the same. The differences between the series must be accounted for using the remaining variables.

When there are multiple measured data points at a given predicted value (e.g., No NM series data), this means there was no variable that could distinguish between the measured values and that the variation seen is either a measure of the random error in the tests or an inadequacy in the model from not having the necessary variables to fit the data better. It was found that the 30s series could not be fit by models similar to the ones that fit the other data series, so the 30s series was eliminated from most further consideration. The 30s data are also the most 'suspect' since much of the data used was from analyses of three year old samples.

The G_D values at a predicted value of 15% for the SB9-NG series shown in Figure 11 correspond to five different measured values ranging from 12.3% to 17.4%. This best model for the SB9-NG series could not distinguish between these data points that were all at 100% AS. Although these runs were at several different PRA values, Table 15 shows that the glycolate destruction does not correlate with the PRA values. There are no other known variables that could have been considered to account for these

differences. Therefore, these data are likely an indication of the variance or repeatability of the data. The two different G_D values at 20% AS also cannot be accounted for by any variable, so are again an indication of the variance of the data. Similarly, for the BH series shown in Figure 11 (orange diamonds), the spread in G_D values from 10.7% to 13.0% is an indication of the variance.

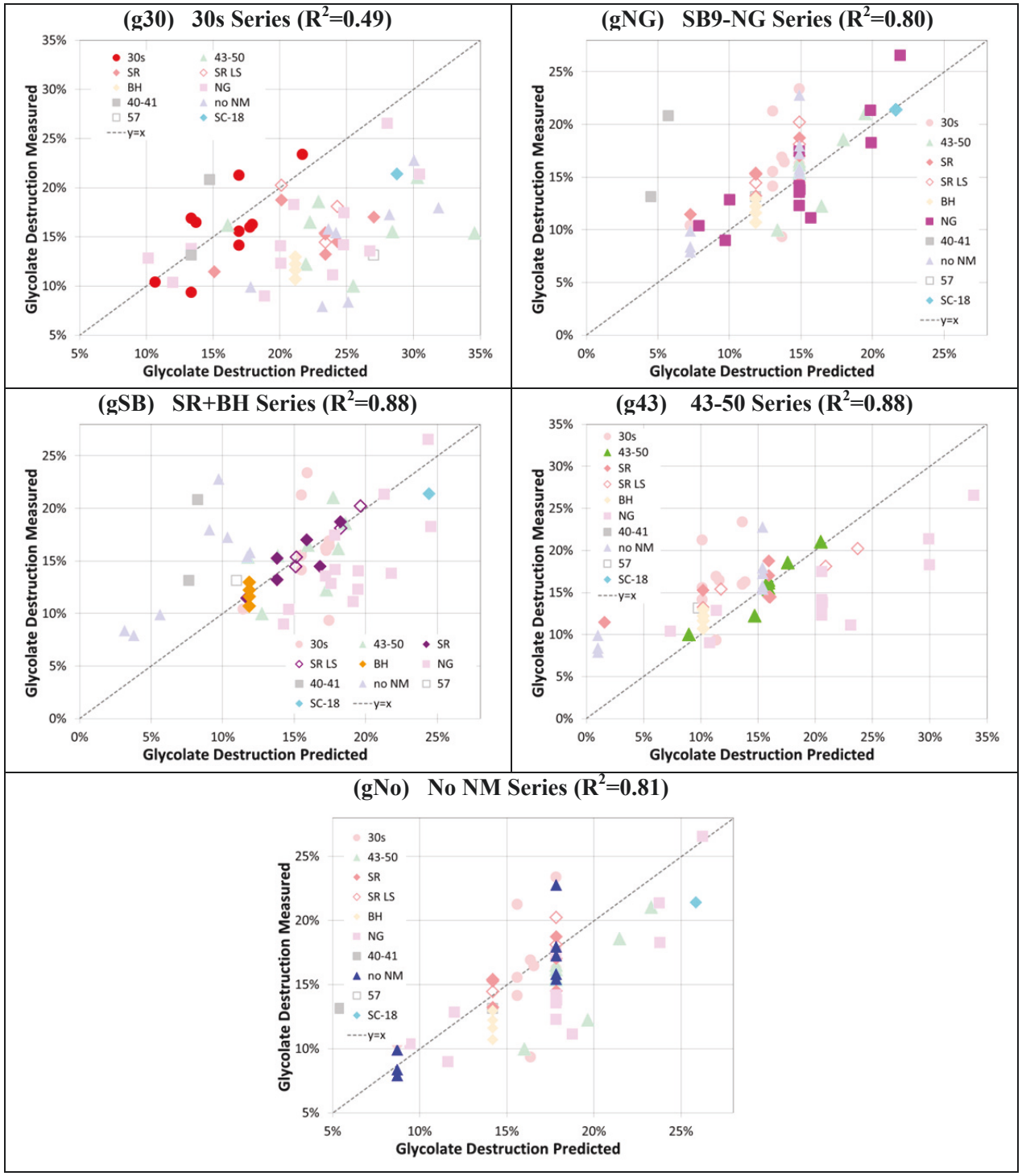


Figure 11. Glycolate Destruction Stepwise Regression of Individual Data Series (MBal Data)

Table 15. Glycolate Destruction for SB9-NG Data at 100% Acid Stoichiometry

Run	Percent Reducing Acid	Measured Glycolate Destruction
NG56	52%	13.5%
NG61	54%	17.4%
NG60	54%	14.1%
NG55	58%	12.3%
NG55A	58%	14.0%
NG57	64%	13.8%

A comparison of the data fits and R^2 values for the MBal data versus the Measured data is shown in Figure 12. The measured data fit for the 30s series has a higher R^2 value due to the high leverage point at about 10%. It is obvious that the R^2 would be less without this influential point. The R^2 values for the other data series are all significantly higher for the MBal data. The MBal glycolate destruction values are also significantly smaller for the 43-50 and 30s series, indicating that glycolate concentrations re-measured by the IC CQ in these older samples was lower than it should have been. Even for the SB9-NG and SR+BH series that were analyzed soon after the tests were completed, the MBal glycolate destruction values are somewhat lower than the measured values. These results indicate that even timely analyses for glycolate appear to result in underestimated values.

The No NM series was left out of further consideration because the chemistry of glycolate destruction without noble metals and mercury appears to be significantly different. The remaining series (SR+BH, 43-50, and SB9-NG) were combined in pairs and all together to see what additional variables would be needed to describe the differences between the data.

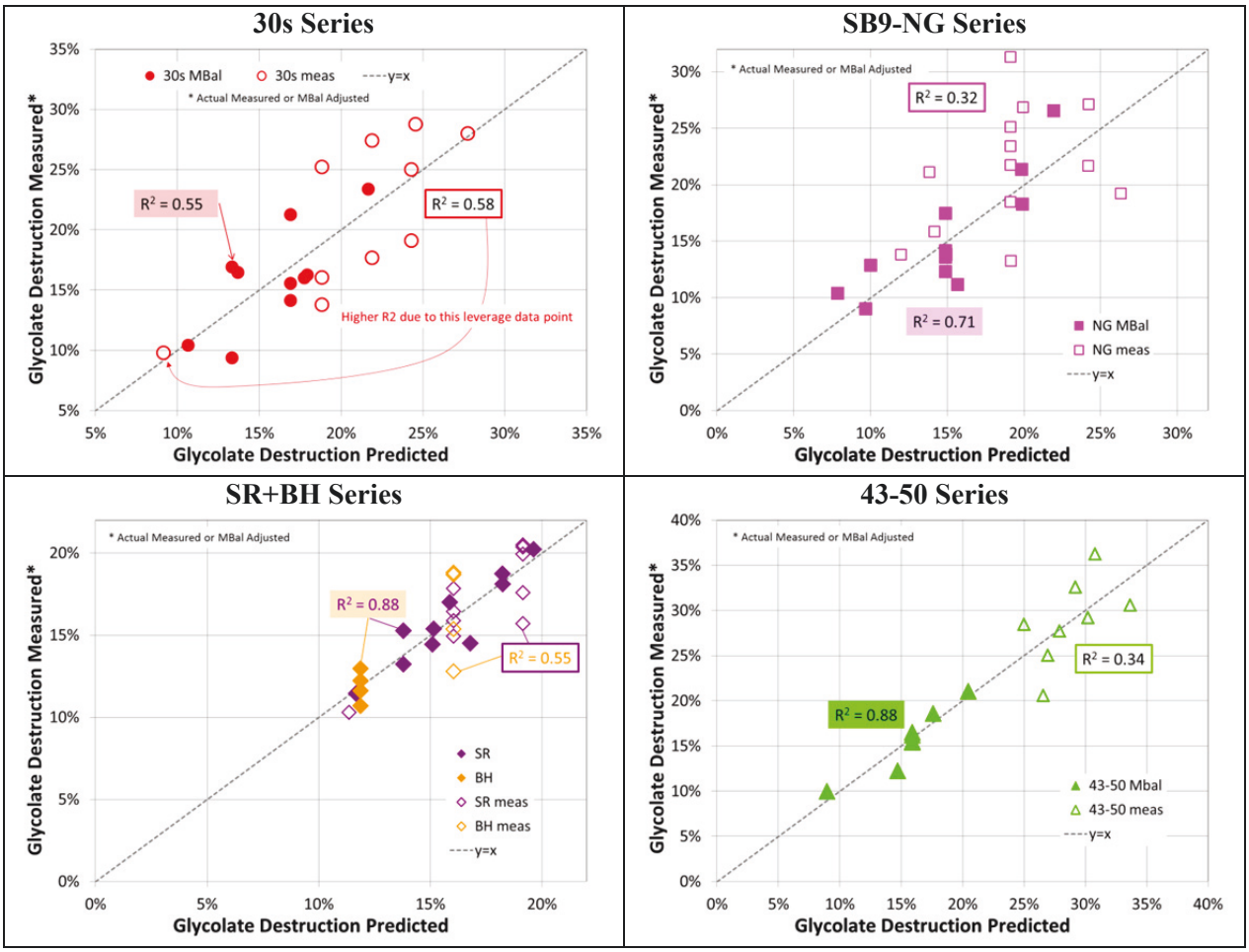


Figure 12. Comparison of Fits of Material Balance (MBal) and Measured (Original) Glycolate Destruction Data

3.4.2 Fitting of Glycolate Destruction Models to Multiple Data Series

The fits of the data series are shown graphically in Figure 13. The R^2 values ranged from 0.73 to 0.80 for the groups of two series. The data set not fit in the group of two was fit well except for the fit of group A, where the fit of the SB9-NG data were poor. Table 16 shows the significant variables found by stepwise regression for the groups of two series (A-C) and the group of three series (G) fit to AS, nitrate, HSV, and Hg. HSV and nitrate were significant for groups B & C, and Mn and Hg were for groups A & B where the SR+BH series is included. The fits for the individual series are reproduced for comparison. HSV is significant for the SR+BH and 43-50,57 series, and not for the SB9-NG series. Nitrate was not significant in any of the individual series because it was not varied within the series. Nitrate, or possibly the correlated nitrite, distinguishes between the series except for A. In all cases, the SC-18 data were over-predicted by the models by 4-8%.

The group of series in D was fit to several models with different variables. The stepwise regression of group G found AS, HSV, nitrite, nitrate, Mn, and Hg to be significant, but the 6 variables were found to result in high correlations between the estimated coefficients. Even certain 5 variable models had high correlation; an unexpected linear relationship exists between nitrite, nitrate, oxalate, and Fe that results in correlation (10140 ≈ - 5.6 nI - 11.3 nA - 26.6 Ox + 9200 Fe). One 5 variable model did not have significant correlation. Table 16 also shows the regression results for grouping G for one 3 variable model, three 4 variable models, and one 5 variable model. The fits of these models are shown graphically in

Figure 14. The fits of the G grouping to five different models (G1-G5) is shown in Table 16. The R^2 and adjusted R^2 for the three variable model G1 is the same as G4, indicating that the addition of HSV adds no significance to the fit. The addition of nitrite in G2 brings slight improvement, but nitrite and nitrate are highly correlated. Model G3 with nitrite, nitrate and HSV is also slightly better than G1. The 5 variable model G5 improves R^2 slightly but adjusted R^2 is smaller indicating over-specification. Model G2 fits the SC-18 data best.

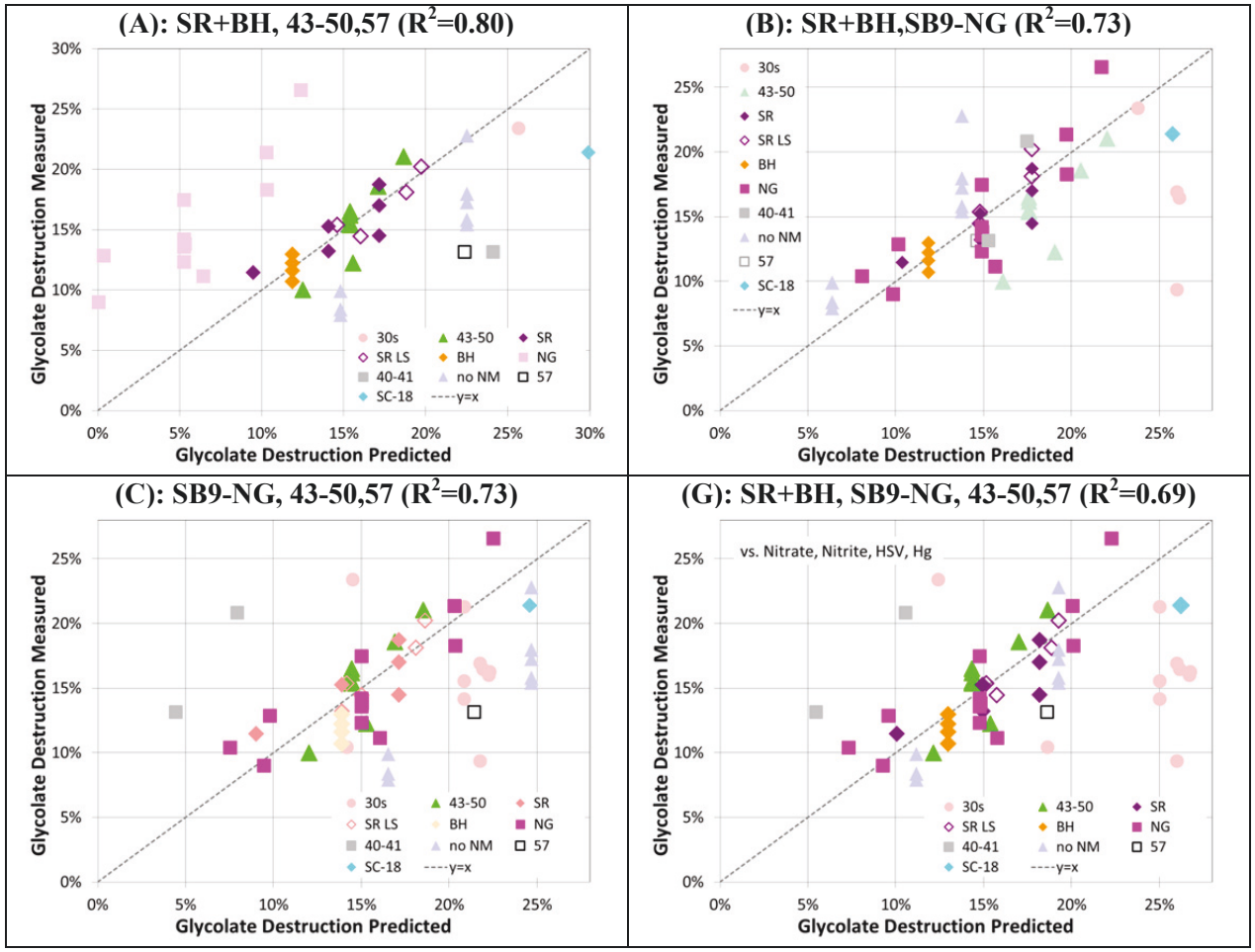


Figure 13. Graphical Display of Fits of Glycolate Destruction

Table 16. Fits of Glycolate Destruction
(see Table 13 for color key)

	A SR+BH, SB9-NG	B SR+BH, 43-50,57	C SB9-NG, 43-50,57	G SR+BH, SB9-NG, 43-50,57
# Data Points	27	23	22	36
# Variables	3	5	3	4
R^2	0.73	0.80	0.73	0.69
R^2 adj	0.69	0.74	0.69	0.66
AS				
PRA				
AS*PRA				
SASV				
HSV		0.05		0.43
nitrite				
nitrate				
oxalate				
Mn				
Hg				0.15
Fe				

	D 43-50	E SR+BH	F SB9-NG
# Data Points	8	14	13
# Variables	2	4	1
R^2	0.88	0.87	0.71
R^2 adj	0.84	0.82	0.68
AS			
PRA		0.11	
AS*PRA			
SASV			
HSV		0.17	
nitrite			
nitrate			
oxalate			
Mn			
Hg			
Fe			

Grouping G (36 Data Points)	(G1) AS nA Hg	(G2) AS nA nI Hg	(G3) AS nA nI HSV	(G4) AS nA HSV Hg	(G5) AS nA HSV Mn Hg
# Variables	3	4	4	4	5
R^2	0.69	0.71	0.70	0.69	0.70
R^2 adj	0.66	0.68	0.66	0.66	0.65
AS					
PRA					
AS*PRA					
SASV					
HSV			0.19	0.43	0.31
nitrite		0.11	0.09		
nitrate					
oxalate					
Mn					0.51
Hg		0.10		0.15	0.12
Fe					

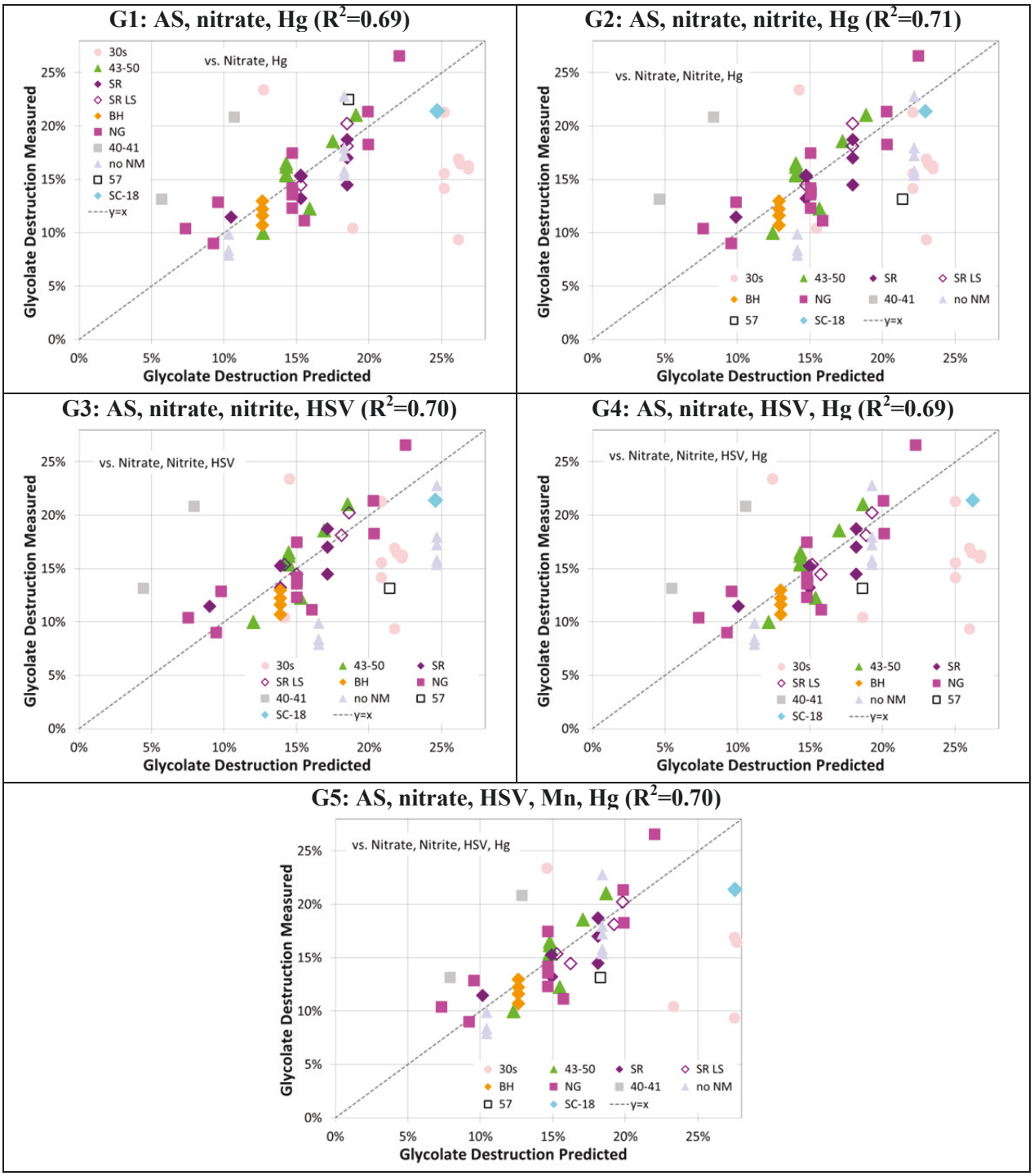


Figure 14. Graphical Representation of Glycolate Destruction for Models G1-G5

In the models G1-G4 shown in Figure 14, in addition to fitting the three data series, models G1 and G4 also fit the No NM data reasonably well, but G2 and G3 do not. None of the models fit the 40-41 series or the 30s series. The predicted glycolate concentrations are compared to the MBal measured values for models G1, G4, and G5 in Table 18. The predicted values range from 95-106% of the MBal values and there is no more than a 2% difference in predicted glycolate concentrations from the three models. The

results show that the 3 variable model predicts essentially the same values as the 4 and 5 variable models. The simplest model should always be chosen. It appears that the variables that influence the glycolate destruction the most are AS, nitrite, and Hg, and that model G1 should be used.

The mostly equivalent fits of the glycolate destruction data in Figure 14 are troubling because the results do not definitively determine what variables are actually important other than AS. Each variable was significant for one or more of the series, and one or more of the groups of two series, but some variables became less significant when the three series were combined. Any of models G1-G5 predict similar glycolate destruction values for the data, so it cannot be determined which model or models would provide the best for the prediction of future tests. Use of the models outside the ranges of the data used to create the models would be extrapolation, and the results of such extrapolation should be used with caution, if at all.

Approximate 95% confidence intervals on the mean and individual values are shown in Figure 15 for the G1 model. The confidence intervals for other fits of the data would be similar. The solid black lines are the 95% confidence on the mean value; e.g., at a predicted G_D value of 15%, for repeated runs, the mean G_D found would be in the range of about 13.8-16.2% (8% relative) with 95% confidence. The dotted black lines show the confidence in predicting the outcome of one run; e.g., at a 15% predicted value, the 95% confidence would be about 10.3-19.8 (32% relative). In other words, if the prediction equation predicts G_D to be 15% for one run, there is a 95% chance that the actual value could range from 10.3% to 19.8%.

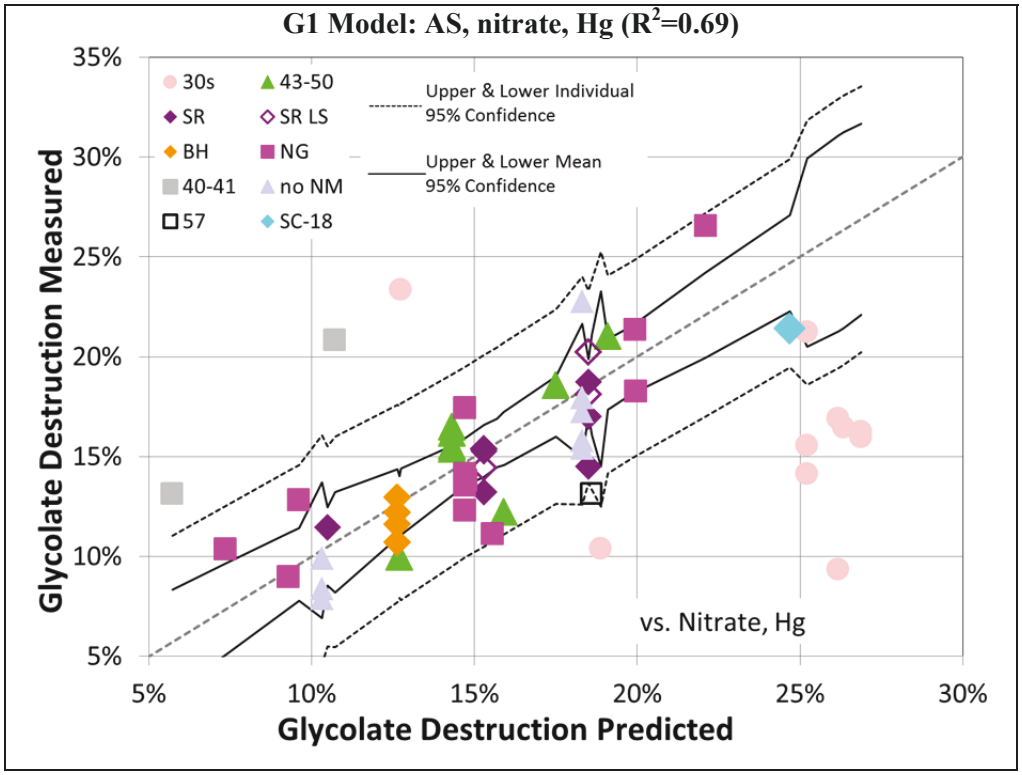


Figure 15. Confidence Intervals on Glycolate Destruction

If a new simulant composition were to be tested, all 5 models could be applied and engineering judgement could then be used to distinguish which values to use if they differ significantly. Similarly, a large scale run with lower HSV should use a model with HSV. Recall that the new flat topped 4-L SRAT vessel has

HSV ~0.65 compared to the old vessel at ~1.1, and DWPF is about 0.40. The parameter estimates in the prediction equations are shown in Table 17. For variables that with P-value significances greater than 0.2 (magenta), the standard error of the parameter values are shown (±).

The same parameter in different models has similar estimates, which is what is expected for reasonable models. Models with drastically different coefficients are suspect because they are not accounting for the effects of variables in the same way.

Table 17. Coefficients in Glycolate Destruction Models G1-G5

(see Table 13 for color key)

Variable	Coefficient in Model					Average
	G1	G2	G3	G4	G5	
R^2	0.69	0.71	0.70	0.69	0.70	NA
Adjusted R^2	0.66	0.68	0.66	0.66	0.65	NA
Intercept	0.300	0.389	0.463	0.331	0.411	0.379
AS	-0.320	-0.322	-0.325	-0.324	-0.319	-0.322
HSV	NA	NA	-0.0244	-0.0178 ± 0.0220	-0.0278 ± 0.0269	-0.0233
nitrite	NA	-8.06E-06	-8.79E-06	NA	NA	-8.43E-06
nitrate	1.92E-05	2.18E-05	2.06E-05	1.94E-05	1.73E-05	1.97E-05
Mn	NA	NA	NA	NA	-0.00876 ± 0.01310	NA
Hg	0.0233	0.0163	NA	0.0171	0.0202	0.0192

Effect on Glycolate Destruction Value			
	Older 4-L	New 4-L	DWPF
HSV	1.1	0.65	0.4
ΔG_D	-2.6%	-1.5%	-0.9%

value ± std. error

(coefficients with all decimal places shown in Appendix B)

The coefficients show that increasing AS decreases G_D (excess dilution effect), increasing HSV decreases G_D (G_D lower in older 4-L tests), and increasing Hg increases G_D (as expected). The negative coefficient on Mn in model G5 is counterintuitive, because more Mn would be expected to cause more glycolate destruction. The high P-value for this coefficient (0.51; see Table 16) indicates it is not very significant, and the standard error in this coefficient is larger than the value itself, also indicating it is not significant. The dependence on HSV is also barely significant. Therefore, model G5 should not be used because it is over-specified. The data in Table 17 clearly show that the additional parameters in models G3-G5 do not significantly improve the fit of the data. Model G1 is the best model that is not over-specified. The equation for glycolate destruction percentage from model G1 is:

$$G_D(\%) = 0.300 - 0.320 * AS\ (\%) + 1.92 \times 10^{-5} * \text{nitrate (mg/kg)} + 0.0233 * Hg\ (\text{wt}\%) \qquad 12$$

(Note that the coefficients with additional decimal places given in Appendix C should actually be used.)

Figure 16 shows several fits of glycolate destruction for the full 56 data points, for all data except the No NM series, and for all data except the No NM and 40-41 series. The R^2 values for these larger series are much smaller because all the series cannot be fit together as well as smaller subsets. Inclusion of the 30s series increases the scatter. Going from 4 to 5 variables does not improve the fit of the 56 data points. The

fit without the No NM series is slightly better. Removing the 40-41 series brings the biggest increase in R^2. Fitting the 40-41 series requires very different values for the coefficients in the models and so forces the model to be quite different than for the data without the 40-41 series.

Overall, it appears that fitting of the 36 data points of SR+BH, 43-50,57, and SB9-NG series gives the best results and uses the most reliable data. The predicted glycolate values for models G1, G4, and G5 are shown in Table 18. The lowest predicted value is 92% of the MBal value and the highest is 106%, showing that the models predict accurate values within ±10%.

Table 18. Comparison of Predicted and MBal Measured Glycolate Values for Models G1, G4, and G5

Run	Glycolate (mg/kg)				G1/MBal	G4/MBal	G5/MBal
	MBal Value	Prediction G1	Prediction G4	Prediction G5			
GN43	53000	51400	51700	51600	97%	98%	97%
GN44	39900	40800	41000	41000	102%	103%	103%
GN45	55900	53500	53900	53800	96%	96%	96%
GN46	48200	48800	49100	49000	101%	102%	102%
GN47	51500	52800	52800	52500	103%	103%	102%
GN48	56700	57900	57900	57600	102%	102%	102%
GN49	46900	47600	47500	47300	101%	101%	101%
GN50	41700	42200	42200	42000	101%	101%	101%
GN57	52600	49300	49300	49500	94%	94%	94%
GN70	50500	50600	50800	50900	100%	101%	101%
GN71	60200	60800	61100	61100	101%	101%	101%
GN72	47300	46400	46600	46600	98%	99%	99%
GN73	51700	50500	50700	50700	98%	98%	98%
GN74	49700	47400	47500	47600	95%	96%	96%
GN75	50300	50300	50500	50500	100%	100%	100%
GN76	49000	50100	49600	49200	102%	101%	100%
GN77	50400	49900	49700	49400	99%	99%	98%
GN78	50200	50300	50400	50300	100%	100%	100%
GN79	46700	46500	46200	46000	100%	99%	99%
GN80	53200	52900	52700	53000	99%	99%	100%
GN81	53600	53000	52800	53000	99%	99%	99%
GN82	53200	53400	53200	53400	100%	100%	100%
GN83	54200	53000	52800	53000	98%	97%	98%
NG51	45300	46100	46100	46200	102%	102%	102%
NG52	65300	67700	67700	67700	104%	104%	104%
NG53	53200	52100	52000	52100	98%	98%	98%
NG54	62900	62600	62600	62700	100%	100%	100%
NG55	69700	67700	67700	67800	97%	97%	97%
NG55A	40500	40200	40100	40200	99%	99%	99%
NG56	56000	55300	55200	55300	99%	99%	99%
NG57	70200	69400	69300	69400	99%	99%	99%
NG58	42700	45200	45100	45300	106%	106%	106%
NG59	45700	47300	47300	47300	104%	104%	104%
NG60	47700	47400	47300	47400	99%	99%	99%
NG61	47900	49500	49400	49500	103%	103%	103%
NG62	56400	53600	53500	53500	95%	95%	95%
SC-18	38400	36700	36500	36200	96%	95%	94%

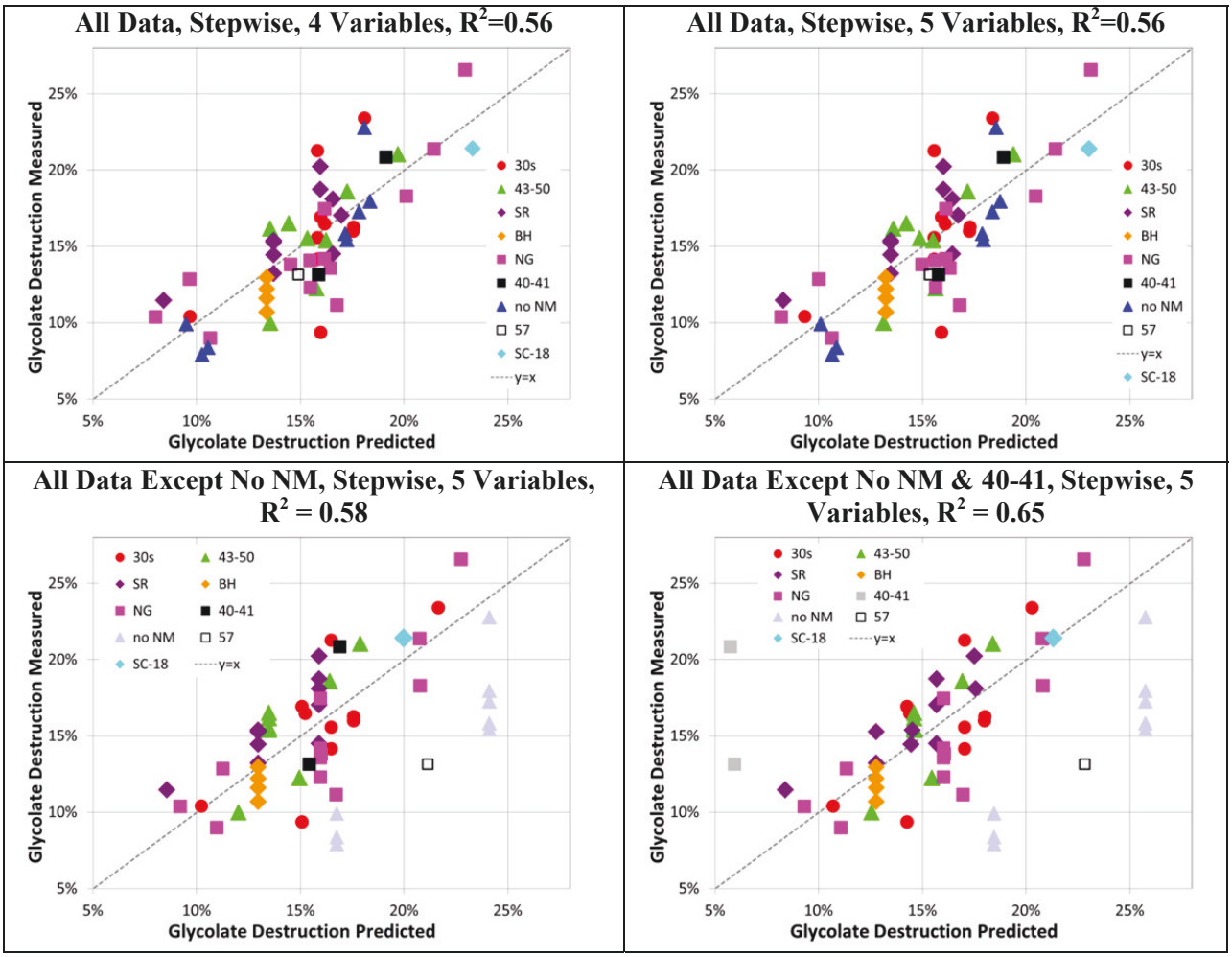


Figure 16. Example Fits of Glycolate Destruction for All Data and Subsets

3.5 Modeling of Glycolate Conversion to Oxalate and Formate

3.5.1 Oxalate

The conversion of glycolate to oxalate may proceed via reactions similar to Reaction 13 shown for reduction of MnO_2:

$$2\ MnO_2 + C_2H_4O_3 + 4\ H^+ = 2\ Mn^{2+} + (COOH)_2 + 3\ H_2O \qquad 13$$

The conversion of glycolate to oxalate was expressed as a percentage as shown in Figure 17. This conversion percentage was fit to the same variables as glycolate destruction with the addition of the noble metals Ru, Rh, and Pd, and whether or not the Ru was added as $RuCl_3$ or $Ru(NO)(NO_3)_3$ (ruthenium nitrosyl nitrate). Ruthenium was added as the nitrosyl nitrate only in the SB9-NG runs. The form of Ru in the SC-18 sludge was assumed to be similar to that generated using $RuCl_3$ in simulant because the oxalate generation was similar. Note it was found that the concentrations of Pd, Rh and Ru were correlated greater than 95%, so each concentration should have the equal ability to predict values. The solid black line in Figure 17 is an example line showing the dependence that would be seen for a constant oxalate conversion at different AS values where the differences are only due to dilution. The data show that the trends are not due to only dilution.

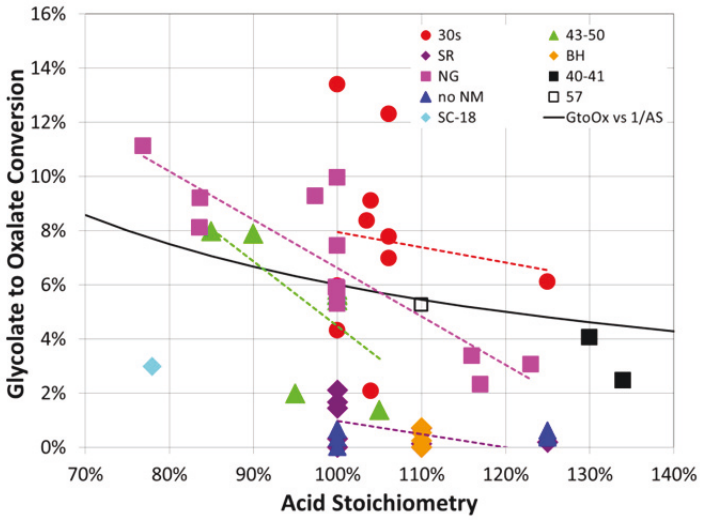


Figure 17. Glycolate to Oxalate Conversion versus Acid Stoichiometry

The data regressed were the series SR+BH, 43-50,57, SB9-NG and SC-18. The 30s series was not included due to significant unexplainable variation in the data. The 40-41 and No NM series were also not included. The data were first fit stepwise versus all of the available variables.

The regression results of fitting several models are shown in Table 19. The best stepwise fit is identified as model X1; the significant variables are AS, nitrite, nitrate, and oxalate. This fit is shown graphically in Figure 18 X1. Several other four variable models (X2-X4) were also fit and gave approximately the same R^2 values. Because a variety of variables could be used to fit the data almost equally well, four variables are significantly over-specifying the model, and a model with less variables should be used. Moreover, nitrite and nitrate tend to be correlated and thus can describe the same dependence. The best three variable model found by stepwise regression was X5. It had an R^2 value similar to the four variable models. However, the best variable in addition to AS and nitrite was Fe, which from a chemical reaction perspective, is not likely to actually be important in this reaction.

Table 19. Significant Variables in Stepwise Fitting of Glycolate to Oxalate Conversion

Data Set	SR+BH, 43-50,57, SB9-NG, SC-18 (37 Points)							
Model Name	**X1**	**X2**	**X3**	**X4**	**X5**	**X6**	**X7**	**X8**
Comment	**stepwise**				**stepwise**	**best 2 var.**	**2nd best 2 var.**	
# Variables	4	4	4	4	3	2	2	2
R^2	0.845	0.845	0.844	0.824	0.833	0.77	0.68	0.64
R^2 adjusted	0.826	0.826	0.825	0.802	0.818	0.76	0.66	0.62
AS								
nitrite								
nitrate		0.09						
Ox								
Mn			0.14					
Fe								
Rh								
Ru-nitrosyl								

(see Table 13 for color key)

The oxalate data were regressed against AS, and then the residual values left after fitting of glycolate to oxalate conversion were fit individually against nitrite, nitrate, and Ru-nitrosyl, as shown in Figure 18 R1-R3. These were the three most significant fits of the residual data. The best two variable model is versus AS and nitrite, shown in Table 19 and Figure 18 X6. The R^2 (0.77) of this model is quite a bit less than the three or four variable models, but is still a reasonably significant fit. Two variable models versus AS and Ru-nitrosyl and AS and nitrate are given by models X7 and X8, respectively, and the fit of model X7 is also shown in Figure 18 X7. Interestingly, a three variable model with AS, nitrite, and Ru-nitrosyl was no better than the two variable model X6. Nitrite and Ru-nitrosyl were found to be significantly correlated, so both can describe the same dependence about equally well.

The recommended model for correlating the glycolate to oxalate conversion is the two variable model X6 with AS and nitrite. The model X7 with the Ru-nitrosyl dependence could also be used as an alternative prediction. Engineering judgement would then be needed to choose which value to use. Fortunately, the amount of oxalate generated is not high, so error in this prediction will not affect the predicted REDOX too much, and the coefficient on oxalate in the REDOX equation (4) is similar to glycolate (6).

The coefficients of the prediction equations for models X6 and X7 are given in Table 20. The equations for models X6 and X7 are given by Equations 14 and 15, respectively.

Model X6:

$$\text{GtoOx} = 0.446 - 0.188 * \text{AS (\%)} - 1.87\times10^{-5} * \text{Nitrite (mg/kg)} \quad (14)$$

Model X7:

$$\text{GtoOx} = 0.218 - 0.171 * \text{AS (\%)} - 0.0187 \langle \text{Ru form} \rangle \quad (15)$$

GtoOx = Glycolate to Oxalate Conversion (%)

where $\langle \text{Ru form} \rangle$ = +1 if Ru nitrosyl nitrate is not present

= -1 if Ru nitrosyl nitrate is present

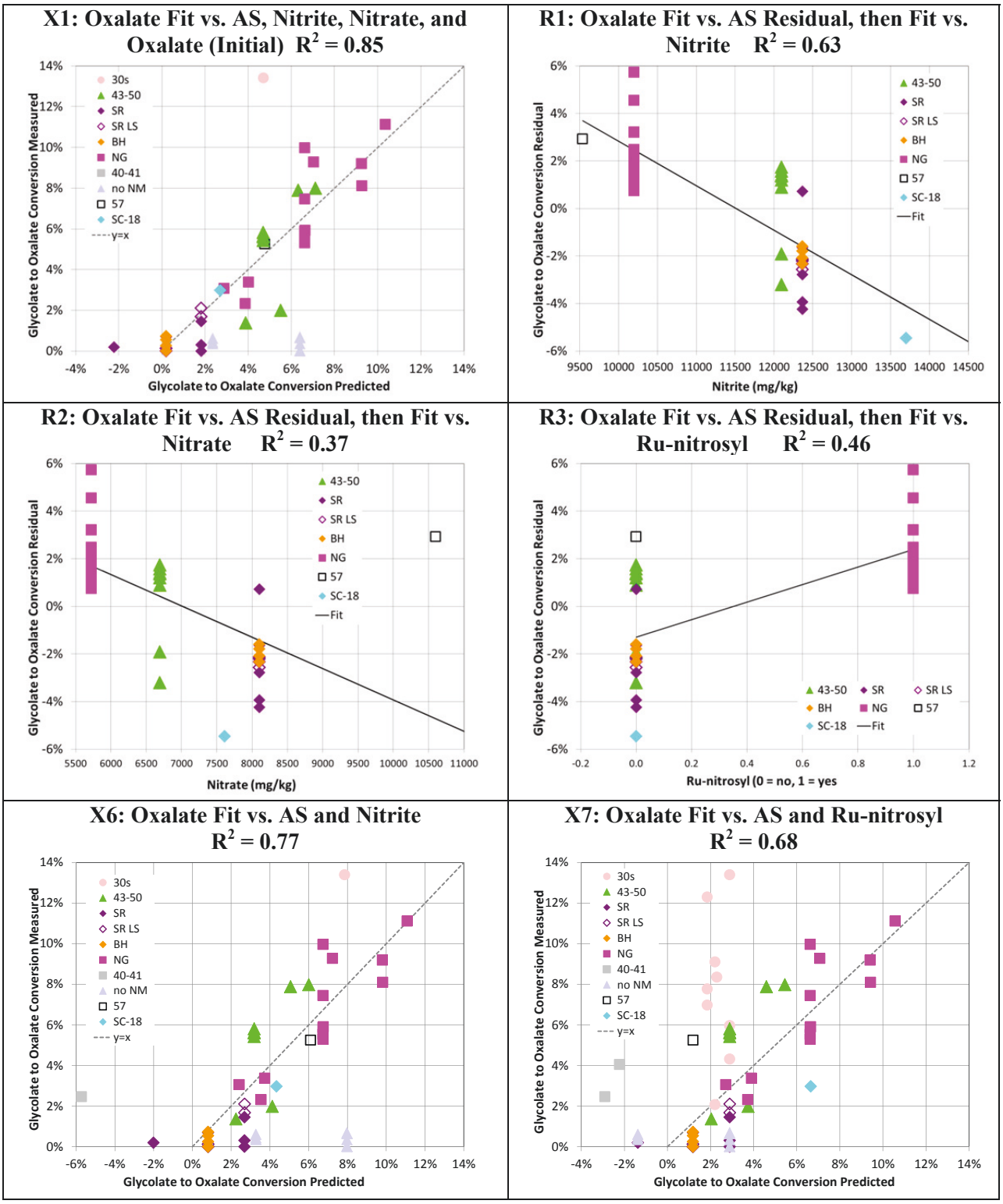


Figure 18. Comparison of Fits of Glycolate to Oxalate Conversion

Table 20. Coefficients in Models X6 and X7

(see Table 13 for color key)

	Coefficient in Model	
Model	**X6**	**X7**
R^2	0.77	0.68
Adjusted R^2	0.76	0.66
Intercept	0.446	0.218
AS	-0.188	-0.171
nitrite	-1.87E-05	NA
Ru-nitrosyl	NA	-0.0187

(coefficients with all decimal places shown in Appendix B)

The graph of glycolate to oxalate conversion model X6 is shown in Figure 19 with the 95% confidence intervals on the mean and individual values shown. At a predicted value of 6%, the uncertainty on the mean is about 5-7% (17% relative) and on an individual test is about 2.5-9.5% (58% relative).

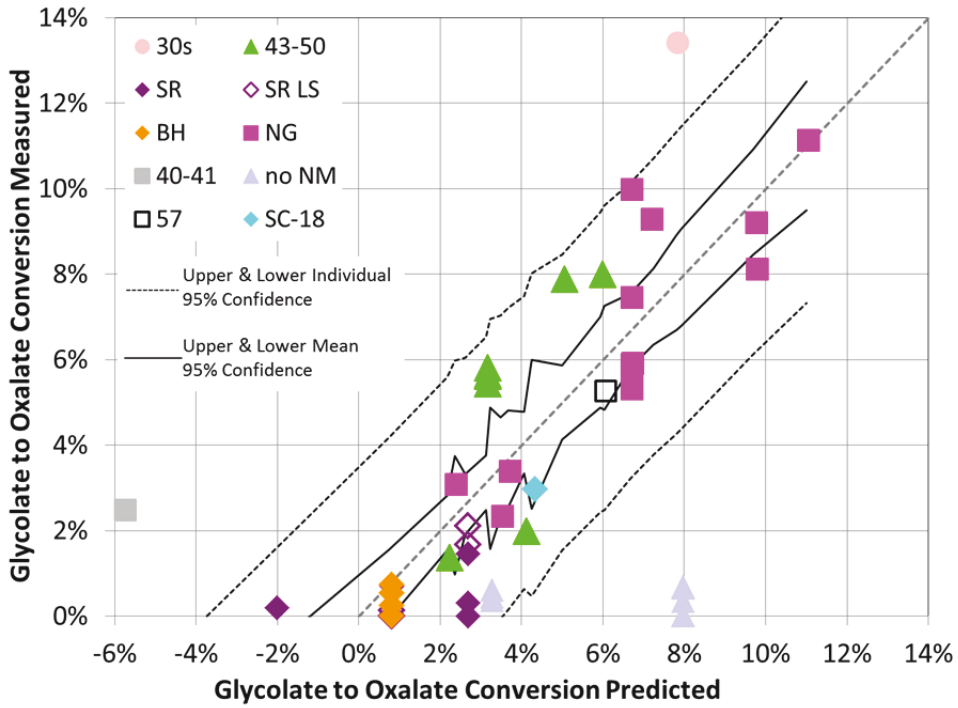


Figure 19. Fit and Confidence Intervals for Glycolate to Oxalate Conversion

3.5.2 Formate

Formate generation from glycolic acid is assumed to occur with simultaneous generation of CO_2 by Reaction 16 where the reduction of MnO_2 is shown for example:

$$2\ MnO_2 + C_2H_4O_3 + 4\ H^+ = 2\ Mn^{2+} + HCO_2H + CO_2 + 3\ H_2O \qquad 16$$

Reduction of 2 moles of MnO_2 by one mole of glycolic acid forms one mole of formic acid and one mole of CO_2 (1:1 ratio). Note that this is an overall reaction and most likely consists of several intermediate steps.

Formate generation from glycolate (GtoF) range from zero to about 7% for simulant tests when noble metals and mercury were present. The radioactive SC-18 run GtoF value was 5.0%. With no noble metals or Hg, the GtoF values are significantly higher and range from about 1% to 29%. These data are shown in Figure 20a-b. The general trend in both the data with noble metals and Hg and the No NM data is that the GtoF values are higher at lower AS. One of the lower No NM data points, at 125% AS, was 6% which is almost as high as the highest value with noble metals and Hg.

These results indicate that the presence of either noble metals or Hg, or both, have a significant effect on the amount of formate in the products. Other similar reactions could also produce formate. Because formic acid is also known to cause reduction of MnO_2, Reaction 17 could also occur:

$$MnO_2 + HCO_2H + 2\ H^+ = Mn^{2+} + CO_2 + H_2O \qquad 17$$

The overall effect of this reaction would be to form less net formic acid and more CO_2, so the formate to CO_2 ratio would be less than 1:1. The simplified chemistry tests also showed that the net formate produced by Reactions 16 and 17 could be significantly reduced by the presence of Hg by Reaction 18:

$$HgO + HCO_2H = Hg^o + CO_2 + H_2O \qquad 18$$

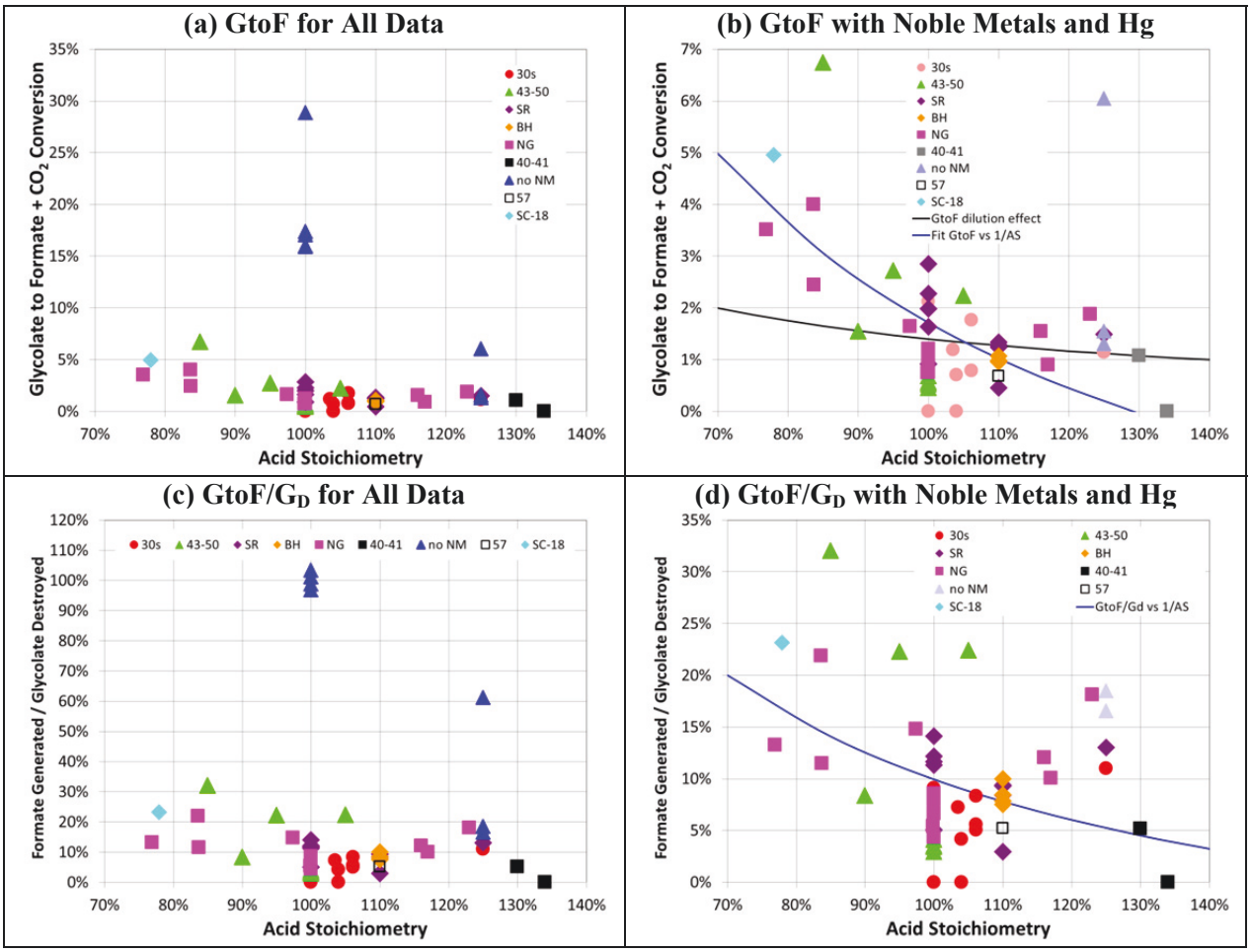


Figure 20. Glycolate to Formate Conversion

Figure 20b shows the data with noble metals and Hg present fit versus the reciprocal of AS (1/AS). (The black line shows how GtoF would vary by dilution only from addition of more glycolic acid at the same absolute conversion of glycolate.) The blue line shows that there is a definite effect of acid stoichiometry on GtoF. The R^2 value for this fit is only 0.44 showing that there is significant variability in the data, but the slope was statistically significant. The GtoF values are definitely higher at the lower AS values.

Figure 20c shows these same data expressed as the ratio of formate generated to glycolate destroyed ($GtoF/G_D$), or the percentage of the reacted glycolic acid that made net formate. With no noble metals or Hg at 100% AS, about 100% of the glycolate destroyed formed formate and CO_2, with no evidence of further destruction of formate (Reaction 17). At 125% AS, this percentage ranged from 12-60%, indicating that at higher AS, there is either some mechanism for destruction of the formate generated or that the amount of formate generated is less. Less formate generation would be consistent with an overall reaction of glycolate with MnO_2 or other oxidant to form only CO_2:

$$3\ MnO_2 + C_2H_4O_3 + 6\ H^+ = 3\ Mn^{2+} + 2\ CO_2 + 5\ H_2O \qquad 19$$

A fit of the $GtoF/G_D$ ratio versus AS, similar to Figure 20b, is shown in Figure 20d. The best model for glycolate to formate conversion with noble metals and Hg is:

$$GtoF\ (\%) = -0.0590 + \frac{0.0762}{AS\ (\%)} \qquad 20$$

3.6 Modeling of Nitrite to Nitrate Conversion as N_C

The nitrite to nitrate conversion data were first fit by individual data series and then in groups of series as was done for glycolate destruction. The 30s and No NM series were both fit poorly by any combination of variables, so these were removed from further consideration in the modeling effort. Table 21 shows the significant variables, R^2 values, and parameter estimate significance for several models for the individual data series. For each data set, the first column is the best stepwise fit, the second column is the fit to a model with AS, PRA, AS*PRA, HSV, and Hg. Note the SB9-NG series has no HSV variation and only the SR+BH series has variation in Hg. The third column for each set has the same variables but without the cross product AS*PRA. The second and third columns correspond to variables that were found to be significant for the groups of two data series.

Table 21. Fit of Nitrite to Nitrate Conversion for Individual Data Series

(see Table 13 for color key)
(Note on abbreviations: e.g., 'n1a' is different than 'N1a')

Series	43-50			43-50,57			SB9-NG			SR			SR+BH		
# Data Points	8			9			13			10			14		
Name **	(n1a)	(n1b)	(n1c)	(n2a)	(n2b)	(n2c)	(n3a)	(n3b)	(n3c)	(n4a)	(n4b)	(n4c)	(n5a)	(n5b)	(n5c)
# Variables	1	4	3	5	4	3	2	3	2	2	4	3	3	5	4
R^2	0.56	0.99	0.88	0.99	0.97	0.87	0.72	0.72	0.71	0.95	0.96	0.96	0.92	0.92	0.92
R^2 adj	0.49	0.97	0.79	0.97	0.94	0.80	0.66	0.62	0.65	0.94	0.92	0.94	0.89	0.87	0.88
AS			0.89	0.20		0.52		0.52			0.81			0.84	
PRA								0.81	0.08		0.95			0.96	
AS*PRA							0.07	0.67			0.96			0.97	
SASV															
HSV								†	†		0.61	0.55		0.68	0.64
nitrite															
nitrate															
Ox															
Mn															
Hg		†	†		†	†		†	†		†	†			
Fe															

** n#a) best stepwise n#b) AS, PRA, AS*PRA, HSV, Hg considered n#c) AS, PRA, HSV, and Hg (if significant)
† no variation in data series

The stepwise regressions show that some combination of AS, PRA, and AS*PRA are significant for every data series. The 43-50,57 series introduced oxalate since Run 57 actually has a different composition than the 43-50 series. In every case, the fit versus AS, PRA, AS*PRA, HSV (where varied), and Hg (SR+BH series) had R^2 values that were the highest or nearly the highest except for the 43-50,57 series. The addition of Run 57 to the 43-50 series proved more problematic for fitting N_C than G_D. Removing AS*PRA lowered the R^2 values for series 43-50 and slightly for series SB9-NG, but raised it for series 43-50,57. The R^2 values for the SR and SR+BH series remained the same and the R^2 adjusted values increased indicating that the addition of AS*PRA was not statistically significant. Therefore, the addition of AS*PRA made little improvement in the fits except for series 43-50 (but not for series 43-50,57). However, including this cross product term probably does not affect the fits, but it does make the P-values insignificant (large), possibly indicating high correlation among AS, PRA, and AS*PRA.

Graphs of the fits are shown in Figure 21. Each data series was fit well by models with the same variables, but the coefficients were significantly different. The fits of series 43-50 and 43-50,57 fit the No NM series reasonably well, but very poorly fit the SR+BH series and especially the SB9-NG series. The SR and SR+BH series fits are extremely good, but neither fit series SB9-NG. Conversely, the SB9-NG series fit did not fit any of the other data series. Note that the SC-18 data point is fit best by the models that include series 43-50. The challenge at this point is to determine which variables can be used to distinguish between these data series.

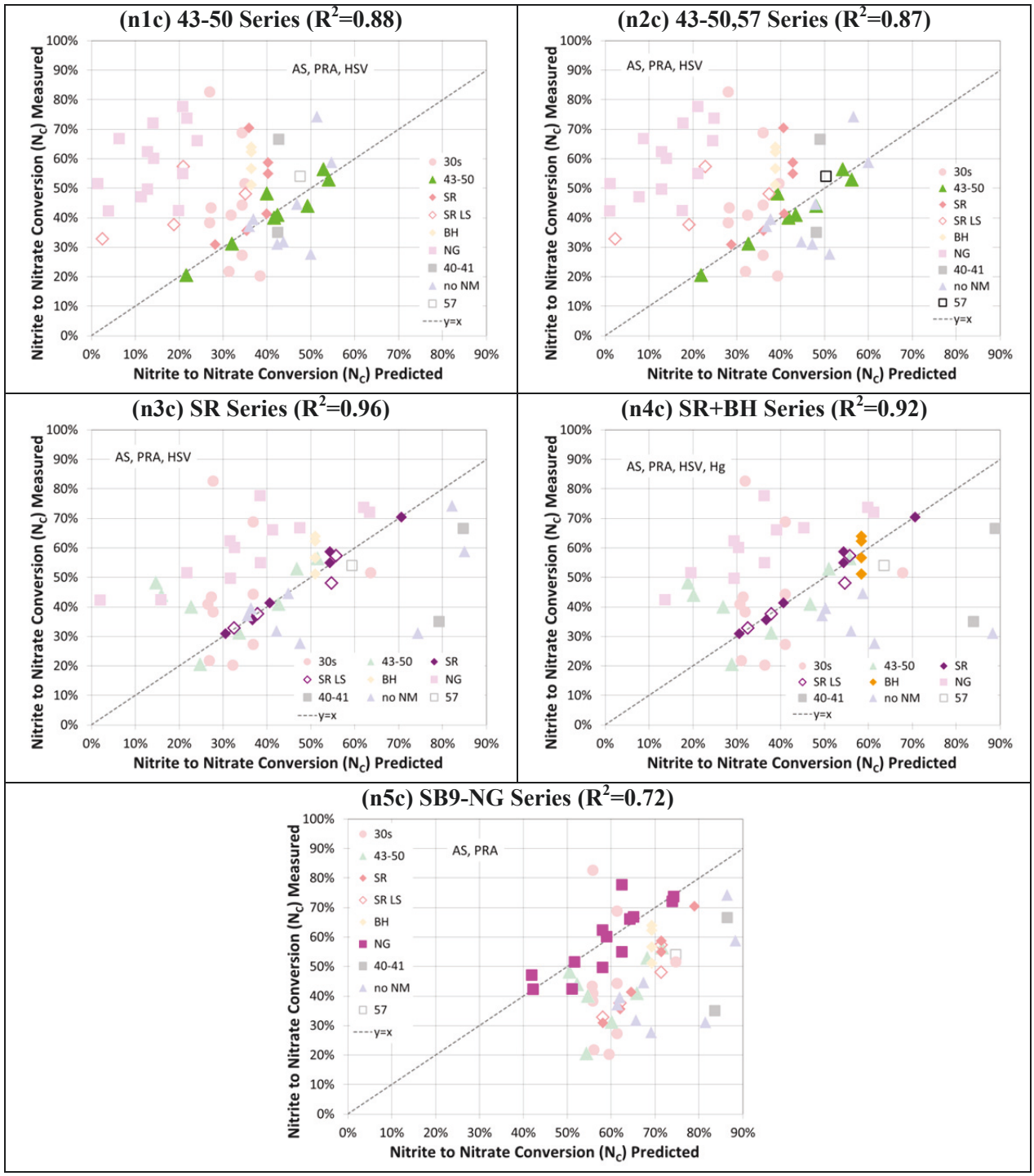


Figure 21. Graphs of Fits of Nitrite to Nitrate Conversion for Individual Data Series

The coefficients in the individual nitrite to nitrate models are shown in Table 22. The dependences on AS between the models are significantly different, especially series 43-50. The inclusion of Run 57 to the series 43-50 data makes the dependence on AS stronger. Recall the 43-50 series was best fit by AS*PRA, which is not included in these models. The opposite signs on HSV would suggest the effect of HSV on nitrite to nitrate conversion is opposite for these simulants; however, the dependence for the SR and

SR+BH series is small and could be considered zero. The difficulty in fitting combined series will be that series 43-50 depends on HSV, but series SR+BH barely does; the combined series model must somehow account for this difference. The most likely outcome is that the combined series model will account for the effect of HSV poorly.

Table 22. Coefficients in Nitrite to Nitrate Conversion Individual Models

(see Table 13 for color key)

Series	SR	SR+BH	43-50	43-50,57	SB9-NG
Name	n4c	n5c	n1c	n2c	n3c
R^2	0.96	0.92	0.88	0.87	0.71
Adjusted R^2	0.94	0.88	0.79	0.80	0.65
Intercept	-0.108	0.0313	0.901	0.769	0.490
AS	1.39	1.39	0.0565	0.216	0.695
PRA	-1.61	-1.61	-1.88	-1.94	-1.04
HSV	-0.0314	-0.0314	0.416	0.429	NA
Hg	NA	-0.0649	NA	NA	NA

The results of fitting pairs of groups of nitrite to nitrate conversion data are shown in Table 23. The first column for each group is the best stepwise regression. The goal for choosing variables to keep for these pairs was for each pair to share as many of the same variables as possible while still being significant.

The N1 grouping of the SR+BH and SB9-NG series is fit by variables common to the individual models: AS, PRA (or AS*PRA), and Hg, but not HSV or SASV. The variable nitrite gets introduced to account for the differences in intercept between the series. For group N1, AS*PRA was removed and the R^2 dropped only slightly from 0.82 to 0.79 (N1b). Removing Hg dropped it more, but the 0.74 R^2 is still reasonable (N1c) for a 3 variable model. Removing nitrite but keeping Hg gave a R^2 of only 0.35 (not shown), so nitrite is definitely needed in the model.

The N2 grouping of the SR+BH and 43-50,57 series stepwise model also contains the common variables AS, AS*PRA, HSV, and Hg. Nitrite and Mn are introduced to account for between set differences. Adding both nitrite and Mn, even though significant, is probably over-specifying the model.

The N2 grouping models all had lower R^2 values than the N1 grouping. Removing Mn and nitrite lowered R^2 some (N2b), and using PRA instead of AS*PRA lowered the adjusted R^2 by 0.01 (N2c). Using nitrite rather than HSV additionally lowered R^2 (N2d). Using neither nitrite nor Hg significantly lowered R^2 (N2e); regression without the Run 57 data gave essentially the same values (N2f). The addition of the fourth variable does not improve adjusted R^2, so the simpler three variable model (N2e) would be preferred for grouping N2. However, model N2d shares the same variables with N1b and therefore could also be considered.

The fitting of the N2 grouping shows the difficulty in using empirical regression for modeling. The models with HSV (N2e), nitrite and Hg (N2d), HSV and Hg (N2c) are approximately equivalent. Therefore, the models cannot definitively determine what variables are truly important. The best model is the one that does not over-specify the variables (N2e), but the less statistically significant nitrite or Hg could be considered for inclusion in the model; nitrite is marginally significant (N2d) and is significant in the N1 models, and Hg is marginally significant in N2c, is significant when nitrite is used rather than HSV (N2d), and is significant in models N1 and N3. This situation shows the difficulty in choosing the appropriate model when there is no significantly better choice.

The N3 grouping of the SB9-NG and 43-50,57 series brings in the common variables AS, PRA, HSV, and SASV (N3a). The dependence on both HSV and SASV is not desired. Also, a very weak dependence on

Fe was introduced, but due to correlation with Hg, Hg could also work. The N3b model shows that removing Fe and SASV and adding HSV and Hg maintained the R^2 value and increased the adjusted R^2 indicating that this model is equivalent or slightly better, and it has fewer variables. (This is a case where stepwise regression does not yield the best model.)

Grouping N3 was found to require both HSV and Hg (N3b, R^2=0.82). Without HSV, the R^2 was 0.70 (N3c) and without Hg it was 0.51 (N3d). Removing Run 57 had no change to the R^2. Therefore, for grouping N3, a four variable model is needed. Note that grouping N3 does not require nitrite as a variable.

The N1, N2, and N3 fits are shown graphically in Figure 22. The fits for the SR+BH and SB9-NG series (N1b) and for the SB9-NG and 43-50,57 series (N3e) are good, but the SR+BH and 43-50,57 series (N2f) fit is not as good. Note that the model in N2e does not fit the SB9-NG series data at all, but the model in N1b fits series 43-50,57 reasonably well. The model in N3b does not fit the SR+BH series.

Table 23. Fit of Nitrite to Nitrate Conversion to Pairs of Series

(see Table 13 for color key)

Name	(N1)			(N2)					
Series	SR+BH, SB9-NG			SR+BH, 43-50,57					
# Data Points	27			23					22
Variables	AS PRA nitrite Hg			AS PRA HSV (Hg)					
Sub-Name	(N1a)	(N1b)	(N1c)	(N2a)	(N2b)	(N2c)	(N2d)	(N2e)	(N2f)
Stepwise?	stepwise			stepwise					
R^2	0.82	0.74	0.79	0.71	0.67	0.67	0.65	0.65	0.66
R^2 adj	0.77	0.70	0.75	0.62	0.60	0.59	0.57	0.59	0.60
# Variables	5	3	4	5	4	4	4	3	3
AS									
PRA	0.16								
AS*PRA	0.09								
SASV									
HSV				0.06	0.17	0.16			
nitrite							0.35		
nitrate									
Ox									
Mn				0.15					
Hg				0.20	0.34	0.34	0.08		
Fe									
Comments		without AS*PRA, Hg	Hg improves fit		without Mn, nitrite	PRA vs. AS*PRA	nitrite vs. HSV	no Hg	without 57

Name	(N3)				
Series	SB9-NG, 43-50,57				
# Data Points	22				21
Variables	AS PRA HSV Hg				
Sub-Name	(N3a)	(N3b)	(N3c)	(N3d)	(N3e)
Stepwise?	stepwise				
R^2	0.82	0.82	0.70	0.51	0.82
R^2 adj	0.76	0.77	0.65	0.43	0.77
# Variables	5	4	3	3	4
AS					
PRA				0.11	
AS*PRA					
SASV					
HSV					
nitrite					
nitrate					
Ox					
Mn					
Hg					
Fe	0.88				
Comments	Fe, Hg highly correlated	without SASV, Hg vs. Fe	HSV significant	Hg significant	similar results without 57

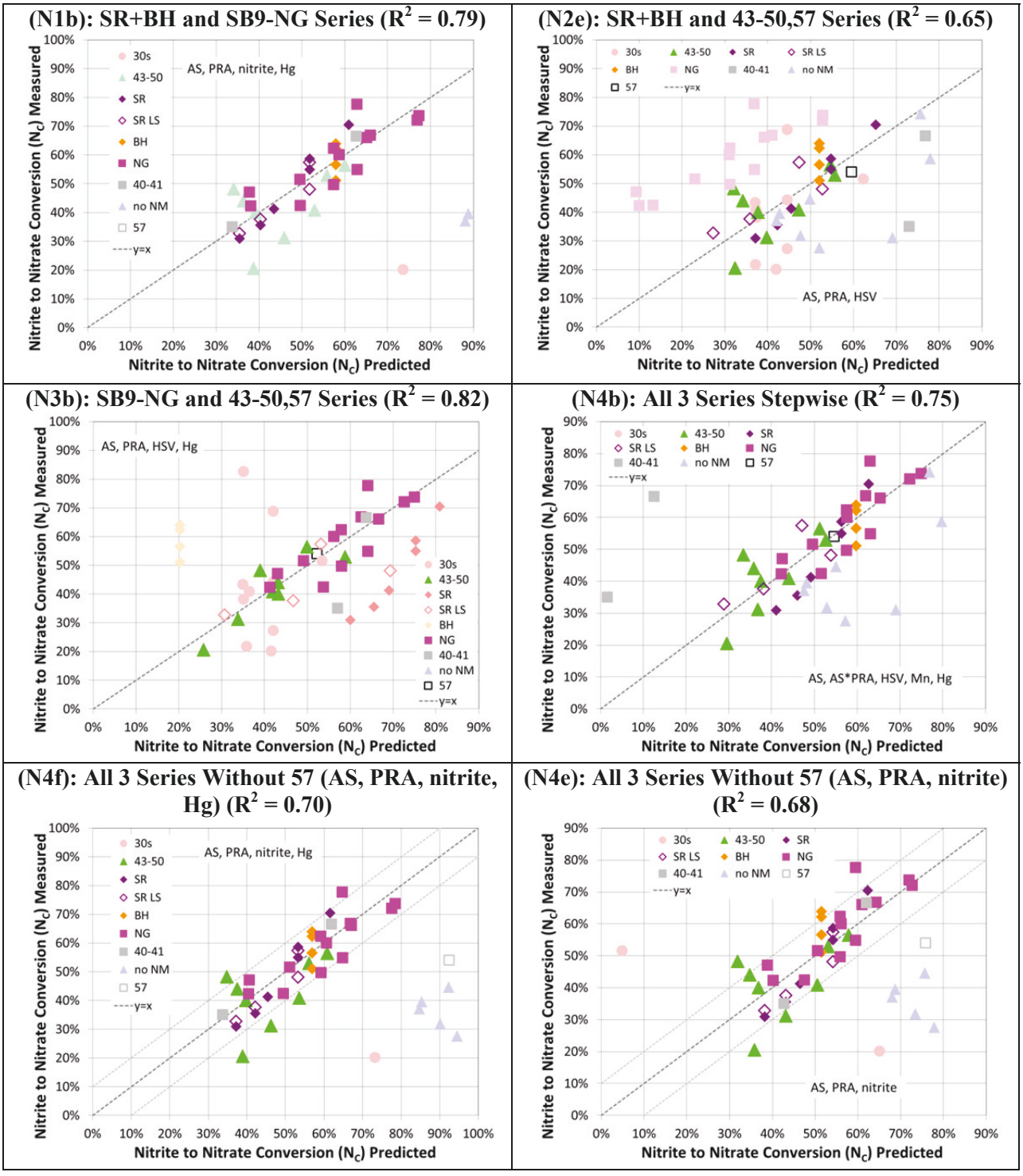


Figure 22. Fits of Nitrite to Nitrate Conversion for Groups of Data Series

The values of the coefficients for these models (N1-N3) are shown in Table 24, along with coefficients for models of the group of three series (N4). The coefficients on AS and PRA are consistent, but the

dependence on Hg between N1 and N3 is of opposite sign and significantly different magnitude. Such differences in a coefficient indicate that the models are not accounting for the effects of the variable in the same way, which is not desired. The effect of Hg on nitrite to nitrate conversion is not likely to be positive for one set of data and negative for another. This type of discrepancy shows the limitations of empirical models. This problem also reinforces the caution about extrapolating the models outside the range of the variables used to create them.

Table 24. Coefficients for Nitrite to Nitrate Models for Groups of Two or Three Series

(see Table 13 for color key)

Name	N1c	N2e	N3b	N4b	N4e	N4f
Series	SR+BH & SB9-NG	SR+BH & 43-50,57	SB9-NG & 43-50,57	All 3 Sets Stepwise	All 3 Sets without 57	All 3 Sets without 57
R^2	0.79	0.65	0.82	0.75	0.68	0.70
Adjusted R^2	0.75	0.59	0.76	0.71	0.65	0.66
Intercept	1.83	0.0483	-0.65	-1.97	1.49	1.83
AS	0.849	0.943	0.641	1.41	0.786	0.802
PRA	-1.28	-1.32	-1.45	NA	-1.33	-1.33
AS*PRA	NA	NA	NA	-1.30	NA	NA
HSV	NA	0.161	0.476	0.198	NA	NA
Nitrite	-1.14E-04	NA	NA	NA	-9.07E-05	-1.11E-04
Mn	NA	NA	NA	0.218	NA	NA
Hg	-0.0768	NA	0.456	-0.0558	NA	-0.0560

(coefficients with all decimal places shown in Appendix B)

The combination of all three series was fit to models (N4) based on the previous results from pairs of series. The two data series model N1b fit the three sets of two data series the best, so it would be expected that the best three series model would include the same variables as N1b.

The significant variables in several fits are shown in Table 25. The stepwise fit of all data is also shown. This fit was very poor, with an R^2 of 0.46, showing it was not possible to fit the data with the 30s, No NM, and 40-41 series included.

Table 25. Fit of Nitrite to Nitrate Conversion for Data Series SR+BH, SB9-NG, 43-50,57

(see Table 13 for color key)

Series	All Data	SR+BH, SB9-NG, 43-50,57				
Name	(N4a)	(N4b)	(N4c)	(N4d)	(N4e)	(N4f)
Comments	stepwise	stepwise			without 57	
# Data Points	56	36			35	
R^2	0.46	0.75	0.40	0.57	0.68	0.70
R^2 adj	0.37	0.71	0.32	0.53	0.65	0.66
# Variables	8	5	4	3	3	4
AS						
PRA						
AS*PRA	0.07		0.16			
SASV						
HSV	0.10	0.06	0.59	no improvement		
nitrite	0.24					
nitrate						
Ox						
Mn	0.13					
Hg	0.11	0.17	0.23	no improvement		0.13
Fe						
Comments	very poor fit to all data		without Mn	nitrite only OK	removing 57 improves	Hg improves slightly

The coefficients for stepwise regression of all three data series are shown in column N4b of Table 24. This fit found AS*PRA to be more significant than just PRA and also included Mn, which was also significant in the stepwise regression of grouping N2. Removing Mn lowered the R^2 to only 0.40. Regression using only the variables chosen for the pairs of series (AS, PRA, HSV, nitrite, Hg) resulted in an R^2 of 0.57. When the Run 57 data point was removed, this same fit gave an R^2 of 0.70 and HSV was no longer found to be significant. Removal of Hg from the model only lowered R^2 to 0.68. Model N4f does indeed have the same variables as N1b.

Graphs of these fitted data series are included in Figure 22. The stepwise fit with the Mn term in N4b fits most of the data reasonably well, but the 43-50 series data are not well fit. The models without Run 57 in N4f and N4e also do not fit the 43-50 series particularly well. Recall that in the two pairs of series groupings with series 43-50 (N2 and N3), the dependence on HSV was significant and that the dependence on Hg was positive. In models N4f and N4e, there is no significant dependence on HSV and the Hg dependence is negative. Therefore, poorer fitting of the 43-50 series is not surprising.

Nitrate concentrations predicted from Table 25 models N4a, N4e, and N4f are shown in Table 26. The predicted values for all three models are between 93% to 111%, except for model N4a for Run NG51, where the predicted value is 139% of the MBal value. The recommended models for nitrite destruction are models N4e or N4f. The values predicted by these two models are not significantly different, with the maximum nitrate concentrations differing by no more than 4%, and typically 2%. Therefore, for prediction of nitrite to nitrate conversion in future tests, it is recommended that both models N4e and N4f

be used to generate predicted values, and that the average of these values be used. These equations to predict nitrite to nitrate conversion are:

$$\text{Model N4e: } N_C(\%) = 1.491 + 0.786 * AS(\%) - 1.33 * PRA(\%) - 9.07\times10^{-5} * \text{nitrite (mg/kg)} \quad (21)$$

$$\text{Model N4f: } N_C(\%) = 1.833 - 0.802 * AS(\%) - 1.33 * PRA(\%) - 1.11\times10^{-4} * \text{nitrite (mg/kg)} - 0.0560 * \text{Hg (wt\%TS)} \quad (22)$$

In the fits without Run 57, the light gray dotted line in these graphs shows ±10% from the y=x line. For model N4e, all of the SR, BH, and SB9-NG series predicted values except two points are within 10% of the measured values. The 43-50 series data have 4 of 8 points beyond ±10%, but still within ±20%. For both models N4e and N4f without fitting Run 57, this data point is significantly over-predicted (open square).

Table 26. Predicted Nitrate Concentrations for Several Nitrite to Nitrate Conversion Models

Run	Nitrate (mg/kg)				N4a/MBal	N4e/MBal	N4f/MBal
		From Table 28					
	MBal Value	Prediction N4a	Prediction N4e	Prediction N4f			
GN43	64900	64000	65600	64900	99%	101%	100%
GN44	54100	52200	52700	52100	96%	97%	96%
GN45	56100	56500	56100	55400	101%	100%	99%
GN46	53500	51400	50600	50000	96%	95%	93%
GN47	54900	57900	58100	57500	105%	106%	105%
GN48	47600	52800	51500	50900	111%	108%	107%
GN49	61900	62900	64600	64000	102%	104%	103%
GN50	69900	67600	70900	70200	97%	101%	100%
GN57	77000	74300	84000	80900	96%	109%	105%
GN70	52600	56000	53800	54000	106%	102%	103%
GN71	67500	66500	65800	66000	99%	97%	98%
GN72	61500	62900	62300	62500	102%	101%	102%
GN73	65500	64800	64400	64600	99%	98%	99%
GN74	56600	59100	57900	58100	104%	102%	103%
GN75	64800	64800	64500	64600	100%	100%	100%
GN76	52200	55200	53000	53200	106%	102%	102%
GN77	64800	64300	64000	64200	99%	99%	99%
GN78	63500	64900	64500	64700	102%	102%	102%
GN79	56000	58000	56800	57000	104%	101%	102%
GN80	62100	62100	62200	61100	100%	100%	98%
GN81	63500	62100	62200	61200	98%	98%	96%
GN82	61100	62100	62200	61200	102%	102%	100%
GN83	63200	62100	62200	61200	98%	98%	97%
NG51	57900	80400	59300	58900	139%	102%	102%
NG52	55000	54600	55100	54600	99%	100%	99%
NG53	49200	49200	48800	48800	100%	99%	99%
NG54	68900	67400	69700	68600	98%	101%	100%
NG55	67100	67900	69200	68500	101%	103%	102%
NG55A	41400	40200	41000	40600	97%	99%	98%
NG56	70400	67900	70600	69400	96%	100%	99%
NG57	56400	56800	56300	56200	101%	100%	100%
NG58	53000	51100	51700	51300	96%	98%	97%
NG59	43200	42800	43800	43200	99%	101%	100%
NG60	58800	54800	56700	55800	93%	96%	95%
NG61	57700	57400	59400	58500	99%	103%	101%
NG62	64200	62700	64300	63500	98%	100%	99%

The graph of the fit in Figure 22 N4e (Table 25 (N4e)) showing approximate 95% confidence bands is given in Figure 23. The 95% confidence on the mean at 50% predicted nitrite to nitrate conversion is about 43-57% (14% relative) and for an individual run it is about 32.5 to 67.5 (35% relative). These values indicate that for multiple runs at the same conditions, the best the mean could be predicted would be about ±14% of the actual value, and that the actual value for a single run could be as much as 35% off.

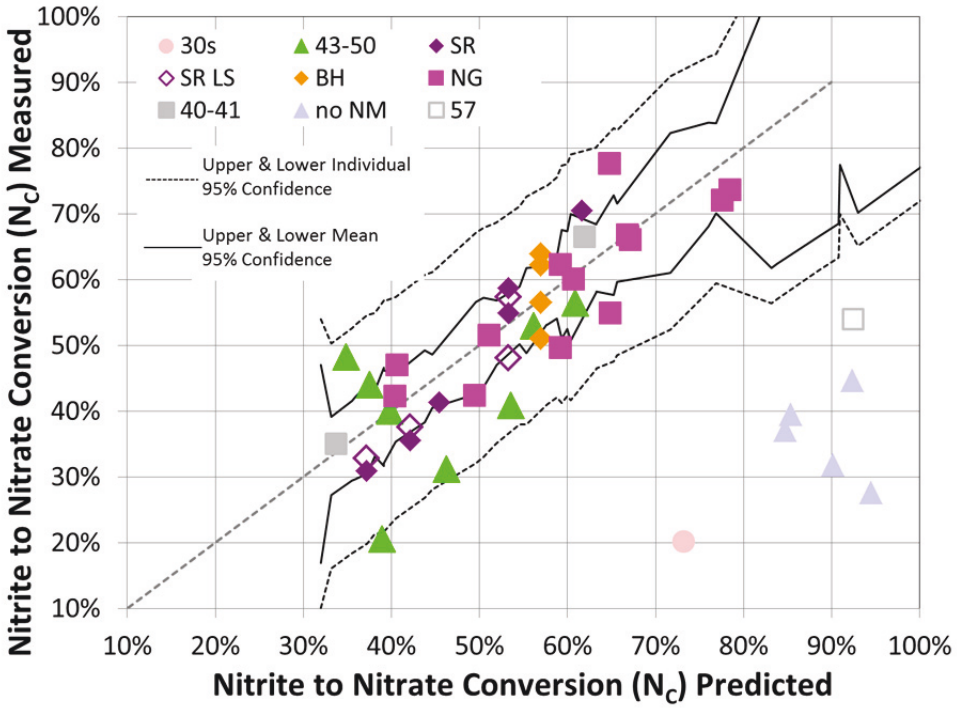


Figure 23. Fit and Confidence Intervals for Nitrite to Nitrate Conversion (Model N4e, without Run 57)

3.7 Combining the Predictions of Glycolate Destruction, Glycolate to Oxalate Conversion, and Nitrite to Nitrate Conversion to Predict Product Compositions and REDOX

The primary need for predicting the product composition in the CPC is to be able to achieve the desired REDOX of the melter feed. To predict the composition, the amount of glycolate destroyed, the amounts of oxalate and formate created, and the conversion of nitrite to nitrate are needed. The REDOX equation inputs are the concentrations of formate, coal, oxalate, glycolate, antifoam, nitrite and nitrate, and Mn. The current REDOX model for the NG flowsheet has the coefficient on Mn set to zero.[16]

REDOX equation:

$$\frac{Fe^{2+}}{\sum Fe} = 0.2358 + 0.1999\left(2[F] + 4[C] + 4[O_T] + 6[G] + 2.88[A] - 5[N] - 0[Mn]\right)\frac{45}{T} \qquad 23$$

where [F] = formate (mol/kg feed)
[C] = coal (carbon) (mol/kg feed)
[O_T] = $oxalate_{total}$ (soluble and insoluble) (mol/kg feed)
[G] = glycolate (mol/kg feed)

[A] = antifoam (mol C/kg feed)
[N] = nitrate + nitrite (mol/kg feed)
[Mn] = manganese (mol/kg feed)
T = total solids (wt %)

Average measured REDOX values for the SB9-NG, SR, and BH series by the 'CC_{hot}' method are plotted versus values predicted from the measured SRAT or SME product compositions, from the compositions adjusted by the MBal method, and from the prediction equations derived in this current work. The "measured" REDOX values used are values tabulated in the references.[16] All tabulated values are used regardless of whether the glass was deemed good and appropriate for the REDOX model. All values are used to demonstrate the improvement in the predicted values using the concentrations adjusted by the MBal method. The use of these REDOX values that were excluded in the REDOX model reports does not imply that these values should be or should have been used for the REDOX models.

In addition, REDOX values determined by the CC_{ramp} method, that has been proven to be inaccurate and unusable for developing a REDOX model, are also used with modification to approximate what the CC_{hot} values would have been. Again, these values are used to demonstrate the improvement in the predicted values using the concentrations adjusted by the MBal method. The use of these REDOX values does not imply that these values should be or should have been used for the REDOX models.

The CC_{ramp} REDOX values for the 30s, 43-50, and No NM series are plotted as the open circles in Figure 24a versus the REDOX predicted from the measured composition values. Note that measured or predicted values less than zero are not shown in these plots. These same data are plotted versus the REDOX predicted from the MBal material balance adjusted compositions as the solid circles. The change in values is shown for two data points by the arrows. This plot shows that the predicted REDOX values using the MBal adjusted concentrations are closer to being equal to the measured values.

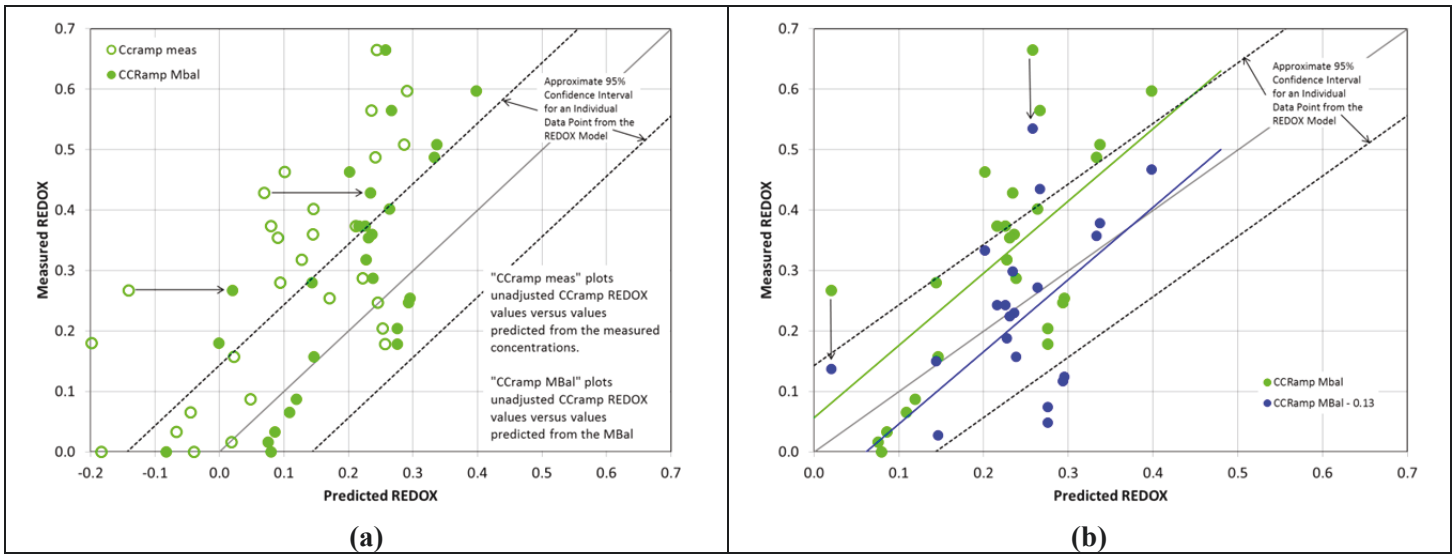


Figure 24. CC_{ramp} REDOX Values Adjusted

To approximate the offset in the CC_{ramp} values versus CC_{hot} values, a constant value of 0.13 is subtracted from every measured REDOX value in Figure 24b. This subtraction does not imply that it is appropriate for REDOX model development to adjust CC_{ramp} values by subtracting a constant.

The SR, BH, and SB9-NG series measured and the adjusted CC_{ramp} REDOX values are plotted in Figure 25a versus the REDOX predicted from the measured concentrations, and versus that predicted by the MBal adjusted concentrations in Figure 25b. These plots show that in general the MBal values fit the

expected correlation where the predicted REDOX should equal the measured, demonstrating that the material balance adjustment to the concentration data is more consistent with the REDOX model in addition to being more consistent with the ability to model the chemistry of the glycolate and nitrite reactions.

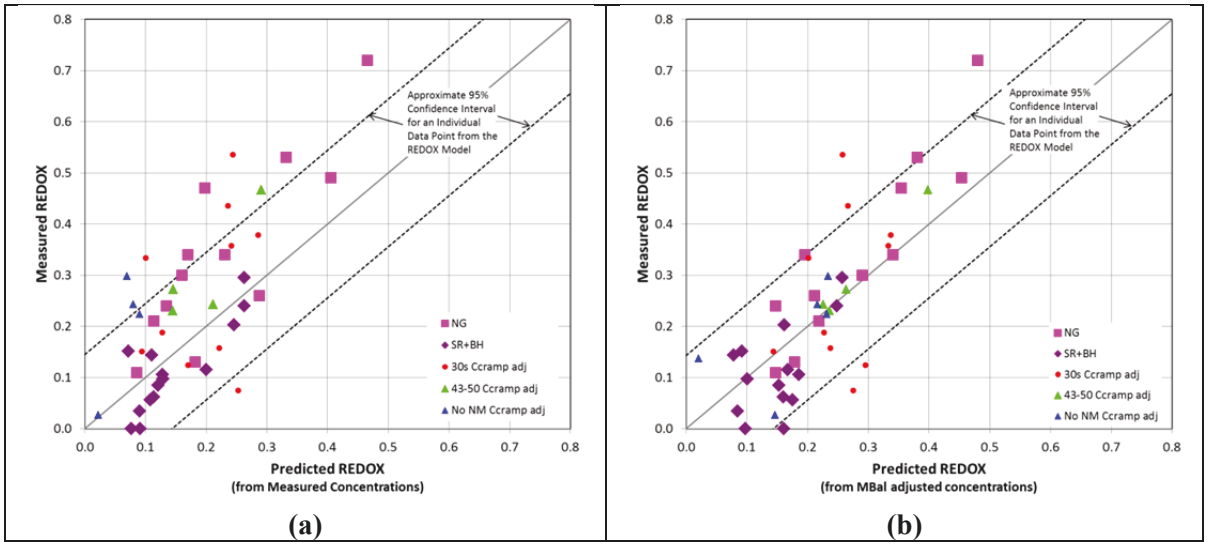


Figure 25. Measured and Adjusted CC_{ramp} Measured REDOX Versus REDOX Predicted from Measured and MBal Adjusted Concentrations

The final test of the chemistry models is to determine how well they predict the REDOX for a test given only the input concentration of the simulant and the proposed acid stoichiometry and percent reducing acid. The models for glycolate destruction (G1), glycolate to oxalate conversion (Table 20 (X6)), and nitrite to nitrate conversion (Table 23 (N2d)) were used to determine the predicted values of these quantities and then determine the corresponding concentrations of glycolate, oxalate, and nitrate in the products for the SR, BH, SB9-NG, and 43-50 series. Glycolate conversion to formate was assumed to be 1.4%. The measured REDOX values for SR, BH, SB9-NG, and 43-50 data series are shown in Figure 26. Note that five 43-50 data points have measured values less than zero and are not shown. The predicted values from the chemistry models, shown in the open symbols, are mostly very close to the values predicted using the MBal adjusted measured compositions.

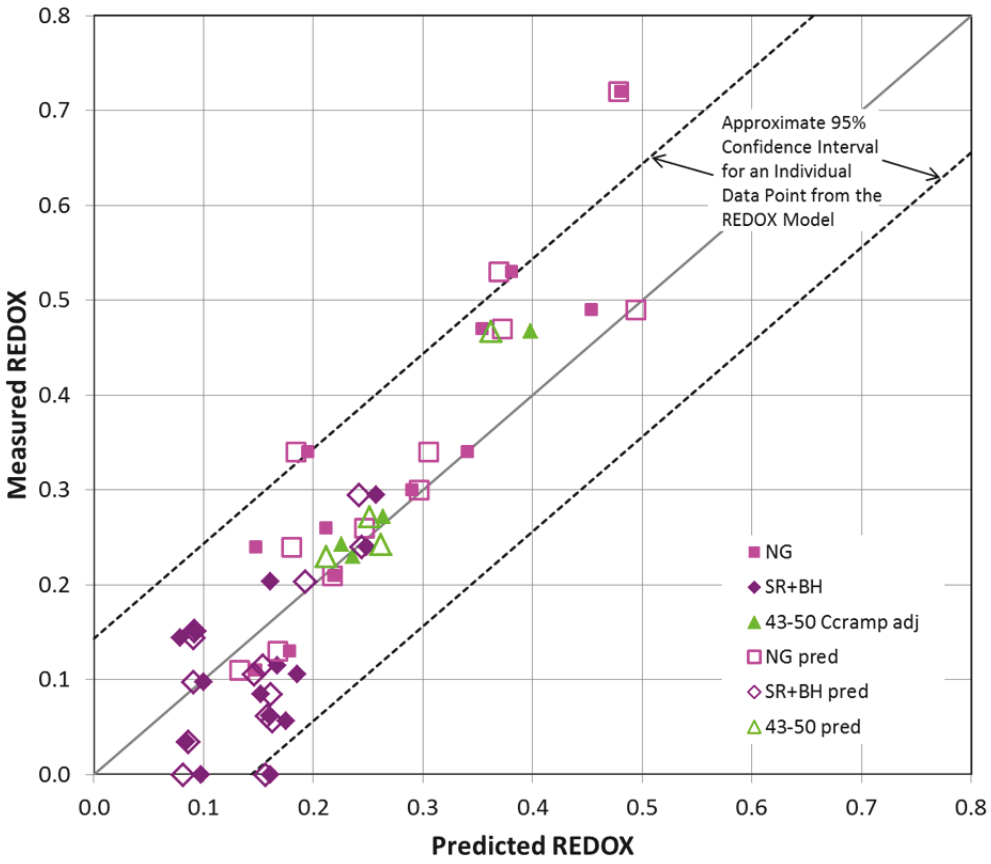


Figure 26. Measured Versus REDOX Predicted from Chemistry Models

To estimate the uncertainties in the product compositions and their effect on hitting the REDOX target, the 95% confidence intervals shown in Figures 15, 19 and 23 were used to determine the uncertainty in the actual REDOX value. The wider 95% confidence intervals on an individual data point were used because the purpose here is to determine how close to the target REDOX the actual REDOX would be for a single test. To do this, the 'worst' values for each concentration were assumed. To get the high REDOX value, the lowest glycolate destruction, highest glycolate to oxalate conversion, and lowest nitrite to nitrate conversion were used. The opposite was done to get the lowest REDOX at a given set of conditions.

The SB9-NG data are plotted in Figure 27 as the predicted versus measured REDOX (opposite of the usual axes), and shows the 95% confidence intervals on an individual test as the vertical bars for each data point. The uncertainty that is shown in the predicted values is actually a measure of the uncertainty in achieving the desired REDOX target using the uncertainty in the chemistry models. The model values are shown by the open squares and are approximately centered in the vertical uncertainty bars. The values predicted from the MBal adjusted concentrations are shown by the solid squares.

Uncertainty in the measured REDOX is not considered in this analysis; it is assumed that if the concentration targets had been achieved, the measured and predicted REDOX values would have been equal. The uncertainty bars show that the uncertainty in the chemistry models results in an uncertainty in the REDOX values that is approximately the same as the ±0.1 REDOX unit uncertainty in the REDOX model. Therefore, it should be possible to use the chemistry models to predict the glass REDOX with similar uncertainty to that inherent in the glass REDOX model. Note that the chemistry model predictions

will be no better than the actual REDOX data; if the predicted REDOX from the actual concentrations does not match the measured REDOX (e.g., NG57), the chemistry model values will be equally in error.

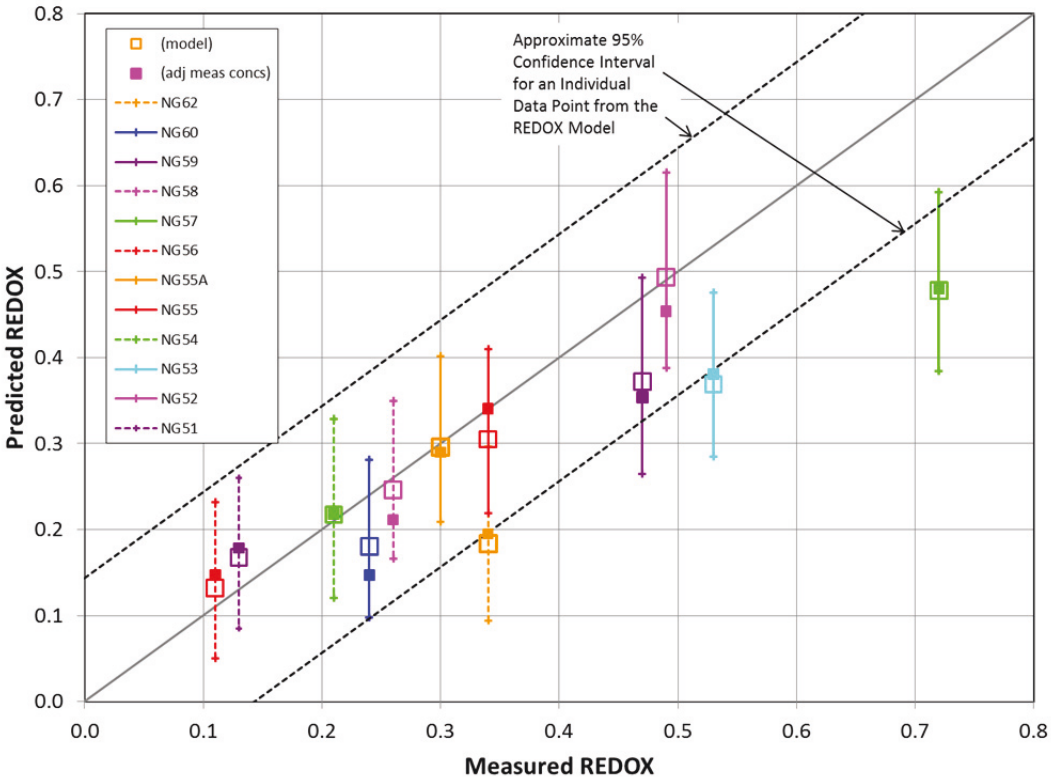


Figure 27. Measured Versus REDOX Predicted from Chemistry Models with Confidence Intervals

3.8 Method for Using the Chemistry Correlation Equations

The acid calculation is an iterative process wherein the PRA value must be guessed and then adjusted until the predicted REDOX matches the target value. In the NF flowsheet, the formate destruction and nitrite to nitrate conversion values were typically estimated from experience and set at constant values. For the NG flowsheet with the chemistry equations, these equations must also be solved simultaneously to predict the glycolate destruction, glycolate to oxalate conversion, and the nitrite to nitrate conversion since these depend on the PRA value. Solving for PRA iteratively will be similar to the NF flowsheet with the addition of these equations.

3.9 Limitations on the Use of the Chemistry Correlation Equations

Use of the prediction equations in DWPF will require further work to determine if the correlations that apply to simulant testing work for actual plant radioactive processing. It was found that the model for glycolate destruction worked for the SC-18 radioactive test, but no conclusions could be drawn for the nitrite to nitrate conversion due to ambiguous nitrate data from this test. It is expected that the equations for DWPF would be similar to those found for simulant testing. Some differences between the simulant testing and actual plant operation that could make the correlations different are:

1. Presence of heels in the SRAT and SME.
2. Additions of Actinide Removal Process products and Strip Effluent.

3. Differences in the detailed timing of acid additions, concentration, and refluxing (e.g., delay times, acid addition rates, boilup rates).

4.0 Conclusions

Note that his current report supersedes the previous work and report[1] on modeling the chemistry of the CPC.

1) The high correlation between some of the potential model variables makes the use of empirical regression somewhat difficult for determining which variables are truly significant.
   a) It is not possible to distinguish between variables if two or more variables have only one value each in two data series, or only two values in several data series. (E.g., data series 1 and 2 have nitrite and Mn both at two concentrations. Differences in response (e.g., glycolate destruction) between the two series could be correlated equally well by either nitrite or Mn.)
2) Adjustment of the SRAT or SME measured glycolate concentrations to close the overall material balance on carbon resulted in glycolate destruction data that were better correlated with the expected variables.
   a) The unadjusted data did not show much statistically significant correlation (very low R^2).
   b) It appears that in some cases, the measured glycolate concentrations are still lower than what appear to be the actual values, possibly by up to about 25%, but on average about 8%.
   c) The correlation between measured and predicted REDOX is better when the adjusted glycolate data are used.
3) The new 4-L CPC testing vessels with the flat metal top heads have head space volume to sludge volume ratios (HSV) values that are closer to DWPF values than the older 4-L vessels. The headspace surface area to sludge volume ratios (SASV) values are still significantly larger than DWPF.
4) The data set from the 30s series was excluded because of high variability in the response data with no apparent variables for correlation. The no noble metals (No NM) data series was also excluded because tests with no noble metals and Hg have no bearing on actual waste testing (but do provide interesting chemistry insights). Data from the 40-41 series that were less washed and very high in oxalate were also excluded from most data analyses. The resulting data used were from three data series and contained 35 or 36 individual data points.
5) With no noble metals and Hg, significantly greater formate is generated from glycolate destruction.
6) The fitting of individual data series for glycolate destruction (G_D) generally correlated with acid stoichiometry (AS), sometimes with percent reducing acid (PRA), and with HSV or SASV if these varied within the data set.
7) Pairs of G_D data series were fit by AS, sometimes PRA and HSV, and also with Hg or nitrate to distinguish between the data series.
8) For all three data series considered (43-50,57; SB9-NG; SR+BH) the best fits of the data were to the variables AS, nitrate, and Hg (model G1).

   $G_D(\%) = 0.300 - 0.320 * AS\ (\%) + 1.92 \times 10^{-5} * \text{nitrate (mg/kg)} + 0.0233 * Hg\ (wt\%)$

9) The glycolate concentrations predicted by the model G1 gave values within 93-106% of the adjusted measured glycolate values, including the SC-18 radioactive test at 93%.

   Therefore, model G1 is recommended.

10) The conversion of glycolate to oxalate appears to depend on AS, and also on nitrite. An somewhat less significant fit of the data to AS and the form of ruthenium was also found, but the data are not conclusive.

    a) Higher conversion to oxalate is correlated with lower AS and with lower initial nitrite.

    b) Higher conversion to oxalate can also be correlated with use of Ru nitrosyl nitrate as the form of Ru in simulant testing. Real waste test SC-18 oxalate conversion was more similar to simulant testing using Ru chloride.

    c) The best model for glycolate to oxalate conversion depended on AS and nitrite, but a dependence on AS and the form of Ru could also be possible. See Equation 14 for additional information on these equations:

    Model X6:

    $$GtoOx = 0.446 - 0.188 * AS\ (\%) - 1.87\times10^{-5} * Nitrite\ (mg/kg)$$

    Model X7:

    $$GtoOx = 0.218 - 0.171 * AS\ (\%) - 0.0187 \langle Ru\ form \rangle$$

11) The generation of formate from glycolate when noble metals and Hg are not present was 100% of the glycolate destroyed (formate generated / glycolate destroyed) at 100% AS and was 12-60% at 125% AS.

12) The glycolate to formate (GtoF) conversion with noble metals and Hg present was less than 7% and increased with decreased AS. The equation describing GtoF is:

$$GtoF\ (\%) = -0.0590 + \frac{0.0762}{AS\ (\%)}$$

13) The conversion of nitrite to nitrate (N_C) for multiple data series was not fit to the variables as well as the glycolate destruction, but the fits to individual data series were significantly better (R^2 from 0.71 to 0.92).

14) The nitrite to nitrate conversion was found to depend on AS, PRA or AS*PRA, HSV when there were differences in HSV, and on Hg for the SR+BH data series.

15) The difficulty in fitting multiple data series for N_C was due to finding no variables that described the differences between the data series as adequately as desired.

16) The best two set models depended on AS, PRA, Hg, and either HSV or nitrite.

17) The best model for the (43-50,57; SB9-NG; SR+BH) combined data series depended on AS, PRA, and nitrite with R^2=0.57.

18) Removing only Run 57 from the combined (43-50,57; SB9-NG; SR+BH) series increased R^2 to 0.70 and Hg became significant (model N4f). Without Hg, this fit was only slightly less significant with R^2 equal to 0.68 (model N4e).
    It is recommended that both models N4e and N4f be used to generate predicted values, and that the average of these values be used.
    Model N4e: $N_c\ (\%) = 1.491 + 0.786 * AS(\%) - 1.33 * PRA(\%) - 9.07\times10^{-5} * nitrite\ (mg/kg)$
    Model N4f: $N_c\ (\%) = 1.833 - 0.802 * AS(\%) - 1.33 * PRA(\%)$
    $$- 1.11\times10^{-4} * nitrite\ (mg/kg) - 0.0560 * Hg\ (wt\%TS)$$

19) The 95% confidence intervals on the predicted mean and individual data points are:

Conversion	Range	95% Confidence on Mean (absolute)	95% Confidence on Individual Value (absolute)
Glycolate Destruction (%)	8-25%	±1.2%	±4.8%
Glycolate to Oxalate Conversion (%)	0-6%	±1%	±3%
Nitrite to Nitrate Conversion (%)	30-80%	±7	±17.5

20) The glass REDOX predicted using the models to calculate the concentrations in the SRAT product has approximately the same uncertainty as the uncertainty in the REDOX model. Both give predicted values for REDOX of about ±0.1 REDOX unit.

5.0 Recommendations and Path Forward for Future Work

1) Future development should include continuing to add simulant and real waste test data to the correlations to improve the ability to predict over a realistic range of sludge compositions.
   a. Consideration should be given to a statistically designed test matrix where the potential variables are varied independently, e.g., vary nitrite while keeping nitrate constant.
   b. Variation of concentrations somewhat outside the normal ranges should be considered so that the data has more leverage.
   c. The range of noble metals concentrations tested should push the boundaries of previous sludge batch and future sludge batch compositions.
2) Further analysis of available data for CPC simulations with Actinide Removal Process and Strip Effluent simulants should be performed to determine what effects extended boiling has on the SRAT product compositions.
3) The Acid Calculation spreadsheet used to conduct CPC simulations should be updated and improved to provide better reconciliation of the actual run data for material balances.
4) Insights from the Simplified Chemistry testing program (SRNL-RP-2014-01183, Rev. 0 Task 4.4)[3] and future work should eventually result in a more realistic acid equation that incorporates the reactions of glycolic acid rather than assuming that formic acid is being used. Regression of data should then be redone with the new acid stoichiometry equation.
5) Prediction of simulant rheology from the input variables of composition, acid stoichiometry, and percent reducing acid should be pursued using the same empirical correlation techniques.

6.0 References

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Appendix A Summary of HSV and SASV Values

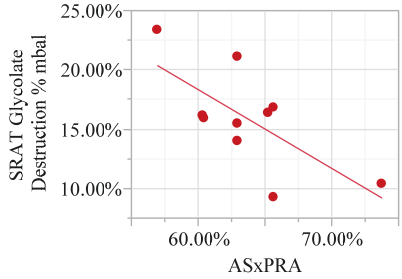
	Total Headspace Surface Area (cm^2)	Sludge Mass (g)	Sludge Volume (mL)	Surface Area to Sludge Volume Ratio (cm^{-1})	Total Headspace Volume (mL)	Headspace Volume to Sludge Volume Ratio
GN34b	1,456	3,174	2,784	0.523	3,323	1.194
GN34c	1,455	3,178	2,788	0.522	3,320	1.191
GN35	1,457	3,169	2,780	0.524	3,327	1.197
GN36	1,456	3,173	2,783	0.523	3,324	1.194
GN36b	1,456	3,173	2,783	0.523	3,324	1.194
GN36c	1,456	3,173	2,783	0.523	3,324	1.194
GN37	1,456	3,174	2,784	0.523	3,323	1.194
GN37b	1,449	3,200	2,807	0.516	3,300	1.176
GN38	1,446	3,209	2,815	0.514	3,292	1.170
GN40	1,421	3,306	2,900	0.490	3,207	1.106
GN41	1,421	3,306	2,900	0.490	3,207	1.106
GN43	1,507	2,980	2,614	0.576	3,493	1.336
GN44	1,507	2,980	2,614	0.576	3,493	1.336
GN45	1,507	2,980	2,614	0.576	3,493	1.336
GN46	1,507	2,978	2,612	0.577	3,495	1.338
GN47	1,382	3,454	3,030	0.456	3,077	1.016
GN48	1,382	3,454	3,030	0.456	3,077	1.016
GN49	1,382	3,455	3,031	0.456	3,077	1.015
GN50	1,382	3,455	3,031	0.456	3,077	1.015
GN51	1,399	3,389	2,973	0.471	3,134	1.054
GN52	1,399	3,389	2,973	0.471	3,134	1.054
GN53	1,400	3,385	2,969	0.472	3,138	1.057
GN54	1,399	3,389	2,973	0.471	3,134	1.054
GN55	1,399	3,389	2,973	0.471	3,134	1.054
GN56	1,399	3,388	2,972	0.471	3,135	1.055
GN57	1,394	3,409	2,990	0.466	3,117	1.042
GN58	1,399	3,388	2,972	0.471	3,135	1.055
GN59	1,399	3,389	2,973	0.471	3,134	1.054
GN70-75	1,380	3,462	3,037	0.454	3,070	1.011
GN76	1,655	20,066	17,602	0.094	6,959	0.395
GN77	1,904	18,094	15,872	0.120	8,689	0.547
GN78	12,378	130,946	114,865	0.108	101,994	0.888
GN79	11,069	152,930	134,149	0.083	82,710	0.617
BH80-83	1,379	3,464	3,039	0.454	3,069	1.010
SB9-NG-51-61	1,052	3,442	3,068	0.343	1,976	0.644
SB9-NG-62	1,002	3,717	3,232	0.310	1,811	0.560
DWPF	284,376	NA	31,570,236	0.00901	12,976,730	0.411

Appendix B Regression Analyses Journal Outputs

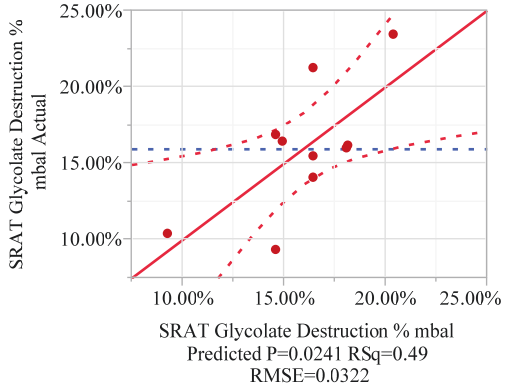
The JMP regression outputs are tabulated here. Each regression is identified as “Exhibit XXX”, where XXX is the shorthand name given to the regression in the tables and figures in the report. All regressions shown in tables and figures are included herein.

The regression results start on the next page.

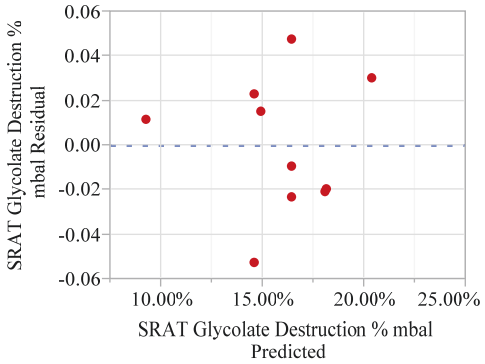
Exhibit g30a: Least Squares Fit Group=30s
Response SRAT Glycolate Destruction % mbal
Whole Model
Regression Plot



Actual by Predicted Plot



Residual by Predicted Plot



Summary of Fit

RSquare	0.490349
RSquare Adj	0.426642
Root Mean Square Error	0.032213
Mean of Response	0.1594
Observations (or Sum Wgts)	10

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	1	0.00798700	0.007987	7.6970
Error	8	0.00830140	0.001038	**Prob > F**
C. Total	9	0.01628840		0.0241*

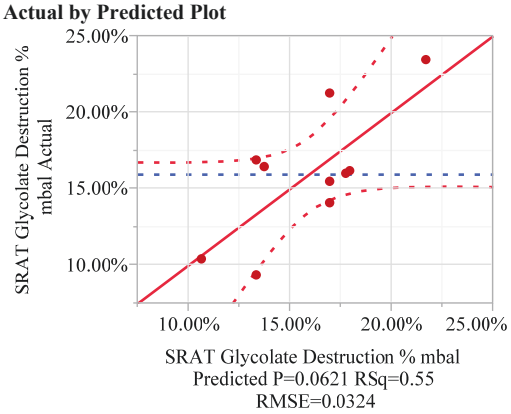
Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	5	0.00258474	0.000517	0.2713
Pure Error	3	0.00571667	0.001906	**Prob > F**
Total Error	8	0.00830140		0.9029
				Max RSq

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.5821304	0.152711	3.81	0.0051*
ASxPRA	-0.664327	0.239454	-2.77	0.0241*

Exhibit g30b: Least Squares Fit Group=30s
Response SRAT Glycolate Destruction % mbal
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.54793
RSquare Adj	0.418767
Root Mean Square Error	0.032433
Mean of Response	0.1594
Observations (or Sum Wgts)	10

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	2	0.00892490	0.004462	4.2422
Error	7	0.00736350	0.001052	**Prob > F**
C. Total	9	0.01628840		0.0621

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	4	0.00164683	0.000412	0.2161
Pure Error	3	0.00571667	0.001906	**Prob > F**
Total Error	7	0.00736350		0.9135
				Max RSq

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	1.1974064	0.37601	3.18	0.0154*
Acid Stoichiometry	-0.348141	0.150098	-2.32	0.0534
Percent Reducing Acid	-1.111513	0.522379	-2.13	0.0709

Residual by Predicted Plot

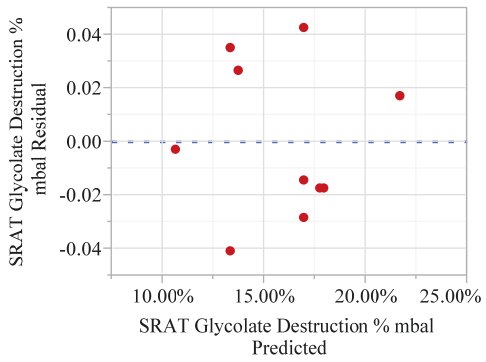
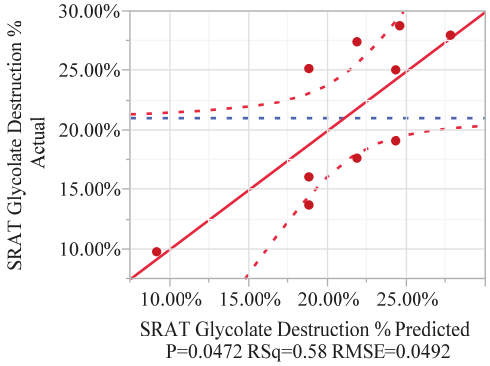


Exhibit g30c: Response SRAT Glycolate Destruction % Group=30s
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.581951
RSquare Adj	0.462509
Root Mean Square Error	0.049215
Mean of Response	0.2105
Observations (or Sum Wgts)	10

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	2	0.02360190	0.011801	4.8722
Error	7	0.01695460	0.002422	**Prob > F**
C. Total	9	0.04055650		0.0472*

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	3	0.00300610	0.001002	0.2874
Pure Error	4	0.01394850	0.003487	**Prob > F**
Total Error	7	0.01695460		0.8332
				Max RSq

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.720416	0.258197	2.79	0.0269*
Acid Stoichiometry	-0.509906	0.23767	-2.15	0.0691
Mn in Feed (before trims) wt % CS	0.0131203	0.009127	1.44	0.1937

Residual by Predicted Plot

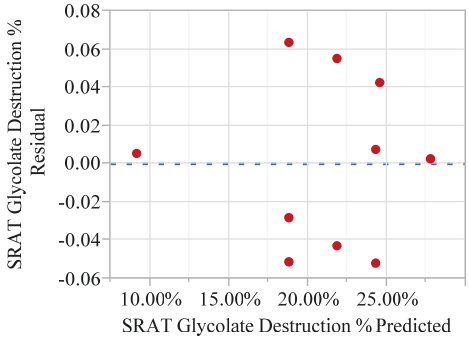
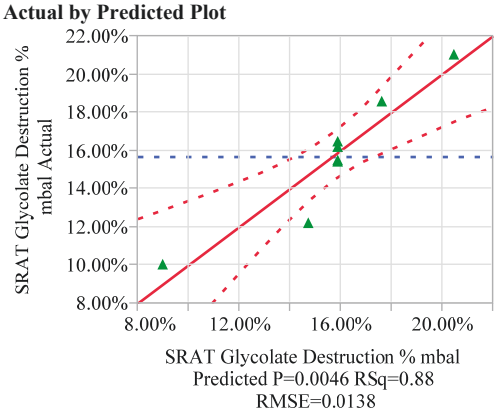


Exhibit g43a: Least Squares Fit Group=43-50
Response SRAT Glycolate Destruction % mbal
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.883755
RSquare Adj	0.837257
Root Mean Square Error	0.013829
Mean of Response	0.15675
Observations (or Sum Wgts)	8

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	2	0.00726933	0.003635	19.0063
Error	5	0.00095617	0.000191	**Prob > F**
C. Total	7	0.00822550		0.0046*

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	2	0.00087017	0.000435	15.1774
Pure Error	3	0.00008600	0.000029	**Prob > F**
Total Error	5	0.00095617		0.0270*
				Max RSq

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.861692	0.117665	7.32	0.0007*
Acid Stoichiometry	-0.574857	0.093499	-6.15	0.0017*
HSV	-0.125946	0.035488	-3.55	0.0164*

Residual by Predicted Plot

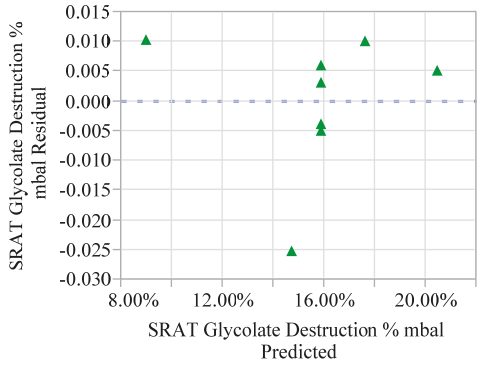
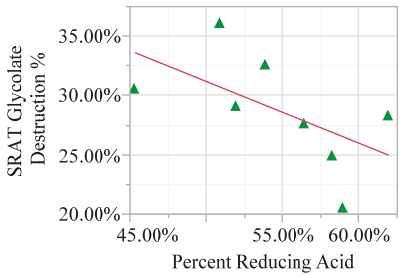
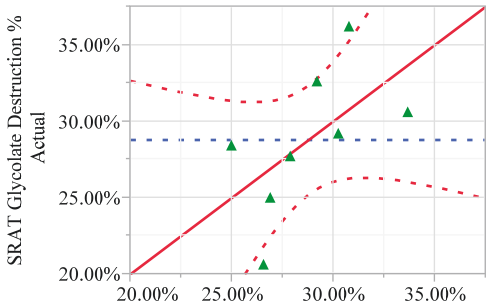


Exhibit g43b: Least Squares Fit Group=43-50
Response SRAT Glycolate Destruction %
Whole Model
Regression Plot



Actual by Predicted Plot



Summary of Fit

RSquare	0.343762
RSquare Adj	0.234389
Root Mean Square Error	0.04126
Mean of Response	0.287875
Observations (or Sum Wgts)	8

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	1	0.00535061	0.005351	3.1430
Error	6	0.01021426	0.001702	**Prob > F**
C. Total	7	0.01556488		0.1266

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.5726102	0.161269	3.55	0.0121*
Percent Reducing Acid	-0.520909	0.293824	-1.77	0.1266

Residual by Predicted Plot

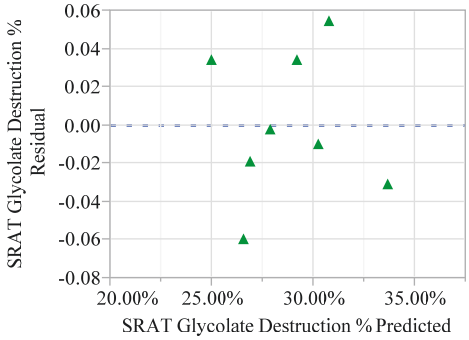
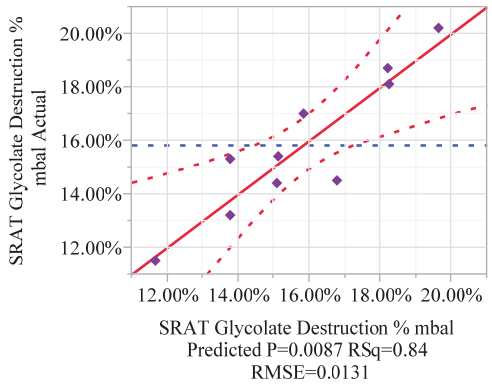


Exhibit gSa: Response SRAT Glycolate Destruction % mbal Data Set=SR
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.838119
RSquare Adj	0.757178
Root Mean Square Error	0.01312
Mean of Response	0.1583
Observations (or Sum Wgts)	10

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	0.00534728	0.001782	10.3547
Error	6	0.00103282	0.000172	**Prob > F**
C. Total	9	0.00638010		0.0087*

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	5	0.00081232	0.000162	0.7368
Pure Error	1	0.00022050	0.000220	**Prob > F**
Total Error	6	0.00103282		0.7034
				Max RSq

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.1900657	0.132858	1.43	0.2025
Acid Stoichiometry	-0.211407	0.057079	-3.70	0.0100*
Percent Reducing Acid	0.3796185	0.186611	2.03	0.0881
SASV	-0.039087	0.024348	-1.61	0.1595

Residual by Predicted Plot

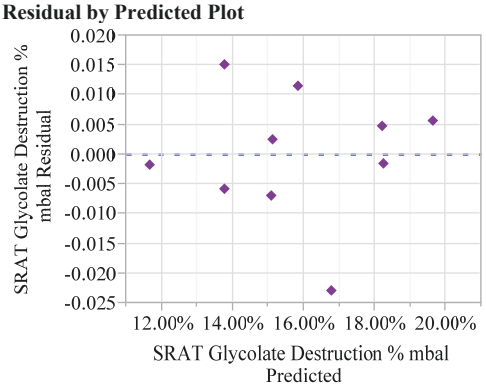
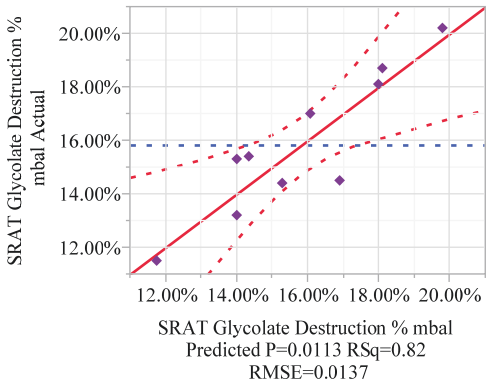


Exhibit gSb: Response SRAT Glycolate Destruction % mbal Data Set=SR
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.822876
RSquare Adj	0.734314
Root Mean Square Error	0.013724
Mean of Response	0.1583
Observations (or Sum Wgts)	10

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	0.00525003	0.001750	9.2915
Error	6	0.00113007	0.000188	**Prob > F**
C. Total	9	0.00638010		0.0113*

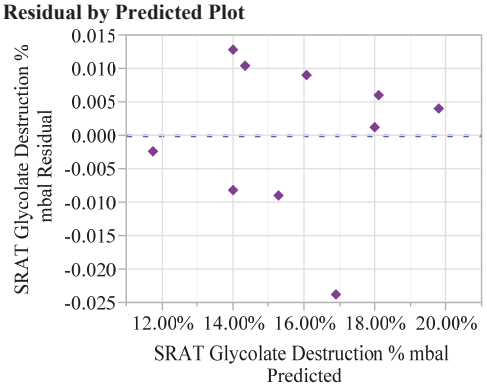
Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	5	0.00090957	0.000182	0.8250
Pure Error	1	0.00022050	0.000220	**Prob > F**
Total Error	6	0.00113007		0.6789
				Max RSq

Parameter Estimates

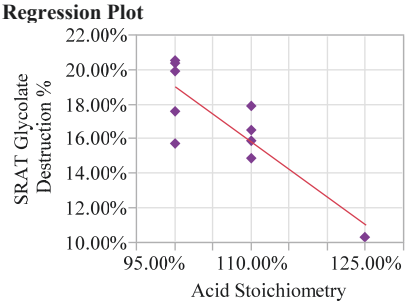
Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.2322842	0.142395	1.63	0.1540
Acid Stoichiometry	-0.211485	0.059914	-3.53	0.0124*
Percent Reducing Acid	0.3227978	0.200634	1.61	0.1588
HSV	-0.027713	0.020436	-1.36	0.2239

Residual by Predicted Plot

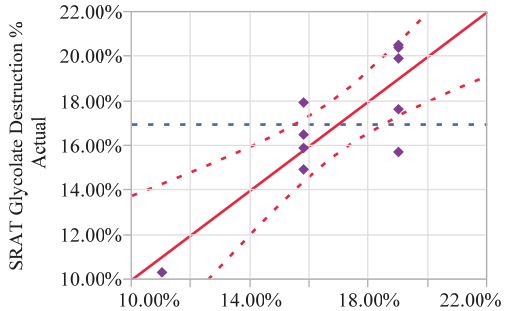


Exibit gSc: Response SRAT Glycolate Destruction % Data Set=SR Whole Model

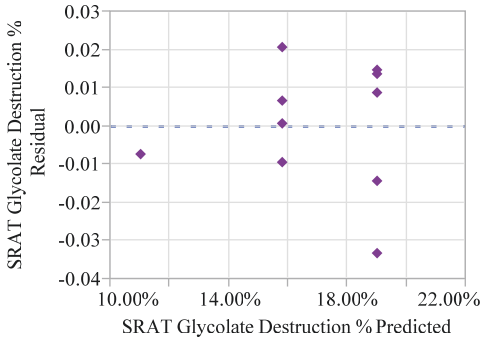
Regression Plot



Actual by Predicted Plot



Residual by Predicted Plot



Summary of Fit

RSquare	0.719612
RSquare Adj	0.684564
Root Mean Square Error	0.017344
Mean of Response	0.1696
Observations (or Sum Wgts)	10

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	1	0.00617600	0.006176	20.5319
Error	8	0.00240640	0.000301	**Prob > F**
C. Total	9	0.00858240		0.0019*

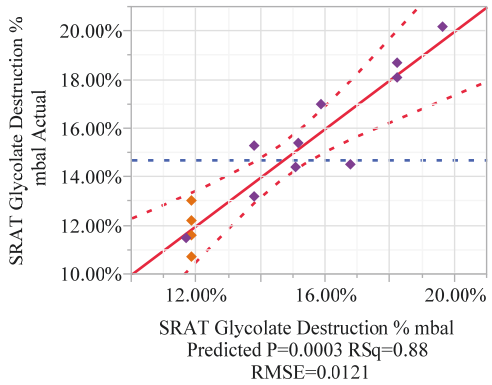
Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	1	0.00016360	0.000164	0.5106
Pure Error	7	0.00224280	0.000320	**Prob > F**
Total Error	8	0.00240640		0.4980
				Max RSq

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.5105768	0.07545	6.77	0.0001*
Acid Stoichiometry	-0.320166	0.070658	-4.53	0.0019*

Exhibit gSBa: Response SRAT Glycolate Destruction % mbal Group=SR+BH
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.881821
RSquare Adj	0.829297
Root Mean Square Error	0.01209
Mean of Response	0.147
Observations (or Sum Wgts)	14

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	4	0.00981643	0.002454	16.7889
Error	9	0.00131557	0.000146	**Prob > F**
C. Total	13	0.01113200		0.0003*

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	5	0.00081232	0.000162	1.2913
Pure Error	4	0.00050325	0.000126	**Prob > F**
Total Error	9	0.00131557		0.4139
				Max RSq

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.1395883	0.125621	1.11	0.2953
Acid Stoichiometry	-0.211407	0.052599	-4.02	0.0030*
Percent Reducing Acid	0.3796185	0.171964	2.21	0.0547
SASV	-0.039087	0.022437	-1.74	0.1155
Hg in Feed wt % TS	0.0235876	0.006955	3.39	0.0080*

Residual by Predicted Plot

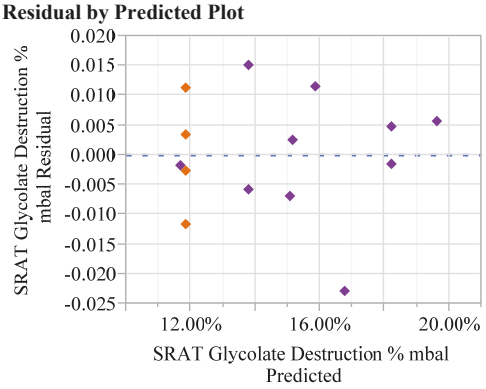
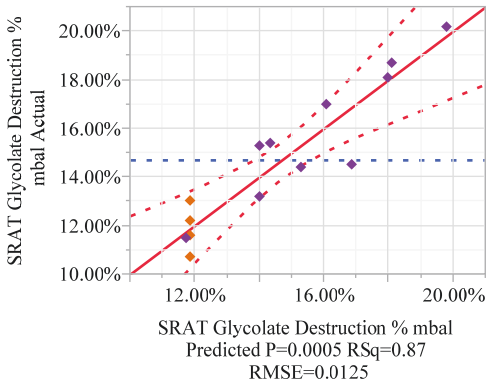


Exhibit gSBb: Response SRAT Glycolate Destruction % mbal Group=SR+BH
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.873085
RSquare Adj	0.816678
Root Mean Square Error	0.012529
Mean of Response	0.147
Observations (or Sum Wgts)	14

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	4	0.00971918	0.002430	15.4784
Error	9	0.00141282	0.000157	**Prob > F**
C. Total	13	0.01113200		0.0005*

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	5	0.00090957	0.000182	1.4459
Pure Error	4	0.00050325	0.000126	**Prob > F**
Total Error	9	0.00141282		0.3713
				Max RSq

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.1797905	0.13426	1.34	0.2134
Acid Stoichiometry	-0.211485	0.054699	-3.87	0.0038*
Percent Reducing Acid	0.3227978	0.183168	1.76	0.1119
Hg in Feed wt % TS	0.0245297	0.007107	3.45	0.0073*
HSV	-0.027713	0.018657	-1.49	0.1716

Residual by Predicted Plot

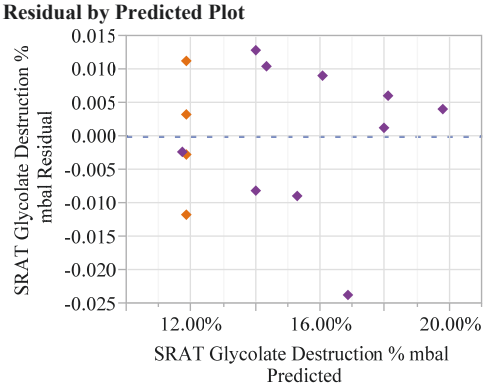
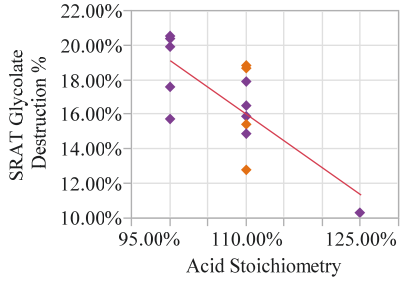
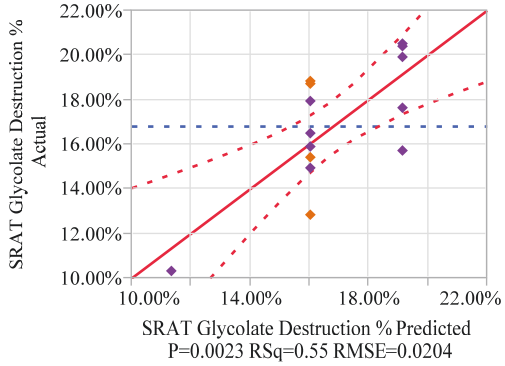


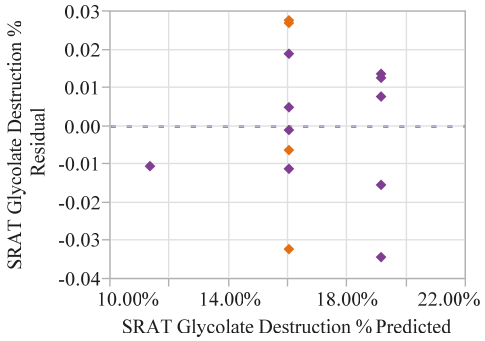
Exhibit gSBc: Least Squares Fit Group=SR+BH
Response SRAT Glycolate Destruction %
Whole Model
Regression Plot



Actual by Predicted Plot



Residual by Predicted Plot



Summary of Fit

RSquare	0.552192
RSquare Adj	0.514875
Root Mean Square Error	0.020412
Mean of Response	0.168071
Observations (or Sum Wgts)	14

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	1	0.00616519	0.006165	14.7972
Error	12	0.00499974	0.000417	**Prob > F**
C. Total	13	0.01116493		0.0023*

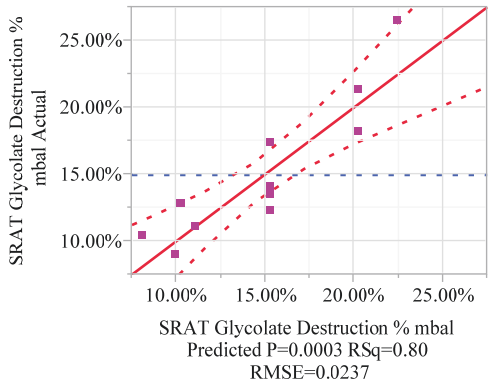
Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	1	0.00025307	0.000253	0.5865
Pure Error	11	0.00474667	0.000432	**Prob > F**
Total Error	12	0.00499974		0.4599
				Max RSq

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.5023754	0.087077	5.77	<.0001*
Acid Stoichiometry	-0.31098	0.080843	-3.85	0.0023*

Exhibit gNGa: Response SRAT Glycolate Destruction % mbal
Group=SB9-NG
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.798507
RSquare Adj	0.758209
Root Mean Square Error	0.023664
Mean of Response	0.149538
Observations (or Sum Wgts)	13

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	2	0.02219150	0.011096	19.8148
Error	10	0.00559973	0.000560	**Prob > F**
C. Total	12	0.02779123		0.0003*

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	4	0.00366440	0.000916	2.8401
Pure Error	6	0.00193533	0.000323	**Prob > F**
Total Error	10	0.00559973		0.1223
				Max RSq

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	-0.068952	0.256592	-0.27	0.7936
Acid Stoichiometry	-0.311963	0.051456	-6.06	0.0001*
SASV	1.5566318	0.747911	2.08	0.0641

Residual by Predicted Plot

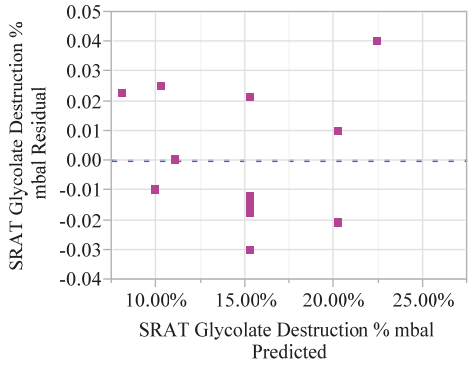
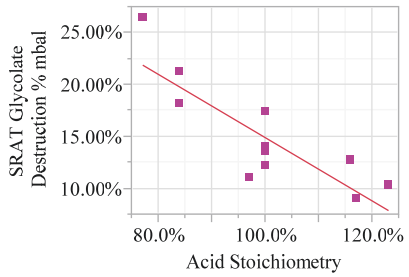
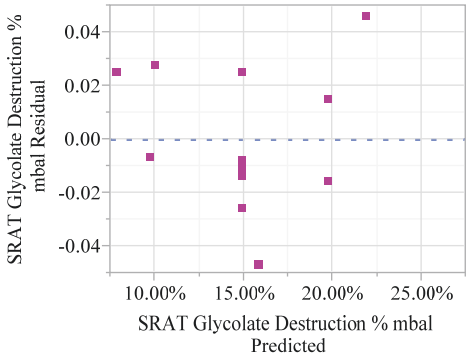


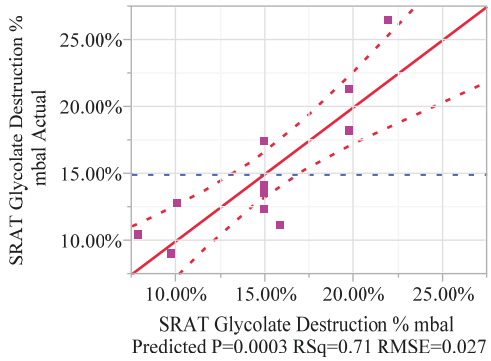
Exhibit gNGb: Response SRAT Glycolate Destruction % mbal
Group=SB9-NG
Whole Model
Regression Plot



Residual by Predicted Plot



Actual by Predicted Plot



Summary of Fit

RSquare	0.711224
RSquare Adj	0.684971
Root Mean Square Error	0.027011
Mean of Response	0.149538
Observations (or Sum Wgts)	13

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	1	0.01976578	0.019766	27.0918
Error	11	0.00802545	0.000730	**Prob > F**
C. Total	12	0.02779123		0.0003*

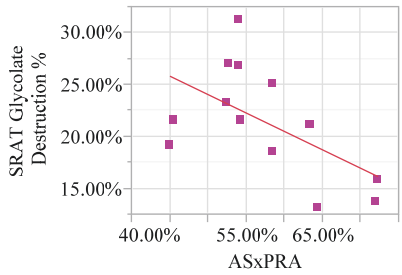
Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	5	0.00609011	0.001218	3.7762
Pure Error	6	0.00193533	0.000323	**Prob > F**
Total Error	11	0.00802545		0.0683
				Max RSq

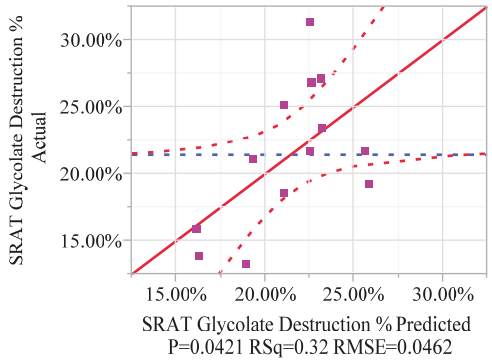
Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.4541473	0.059	7.70	<.0001*
Acid Stoichiometry	-0.305078	0.058613	-5.20	0.0003*

Exhibit gNGc: Least Squares Fit Group=SB9-NG
Response SRAT Glycolate Destruction %
Whole Model
Regression Plot



Actual by Predicted Plot



Summary of Fit

RSquare	0.324528
RSquare Adj	0.263122
Root Mean Square Error	0.046175
Mean of Response	0.214385
Observations (or Sum Wgts)	13

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	1	0.01126797	0.011268	5.2849
Error	11	0.02345311	0.002132	**Prob > F**
C. Total	12	0.03472108		0.0421*

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.4181715	0.089566	4.67	0.0007*
ASxPRA	-0.355145	0.154485	-2.30	0.0421*

Residual by Predicted Plot

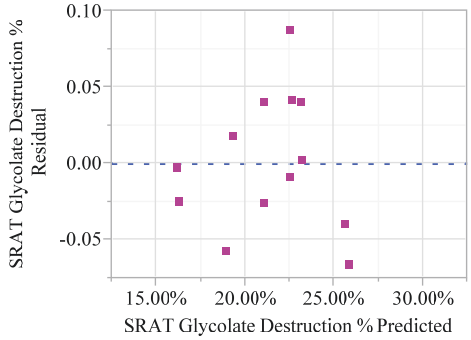
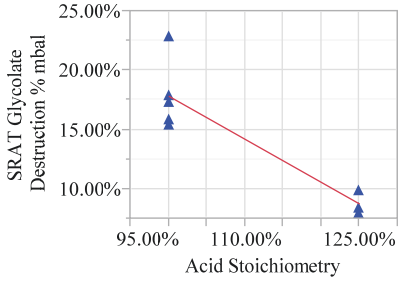
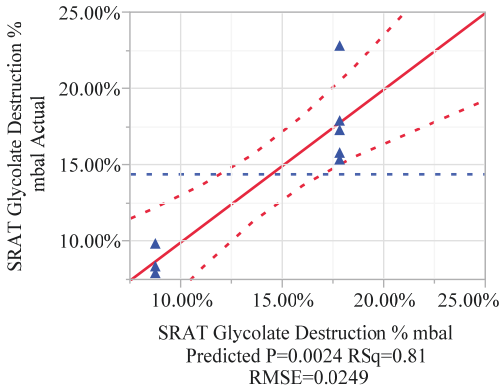


Exhibit gNoa: Least Squares Fit Group=No NM
Response SRAT Glycolate Destruction % mbal
Whole Model
Regression Plot



Actual by Predicted Plot



Summary of Fit

RSquare	0.807039
RSquare Adj	0.774879
Root Mean Square Error	0.024893
Mean of Response	0.14425
Observations (or Sum Wgts)	8

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	1	0.01554963	0.015550	25.0944
Error	6	0.00371787	0.000620	**Prob > F**
C. Total	7	0.01926750		0.0024*

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.5426667	0.080019	6.78	0.0005*
Acid Stoichiometry	-0.364267	0.072716	-5.01	0.0024*

Residual by Predicted Plot

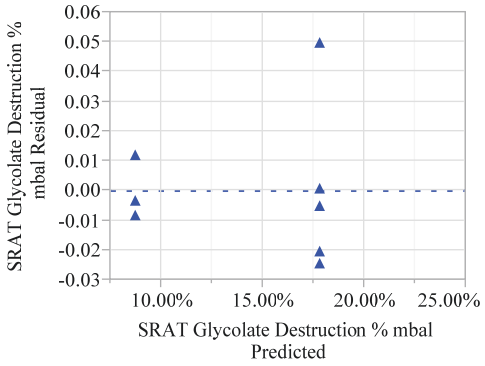
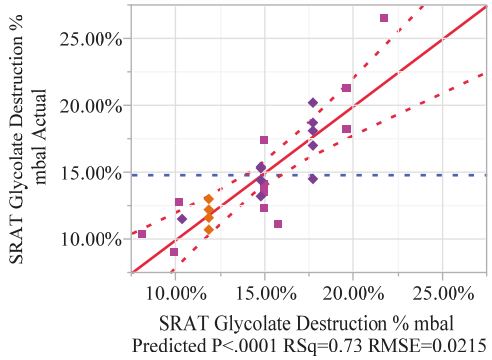


Exhibit A: Response SRAT Glycolate Destruction % mbal JMP Grouping=27 (SR+BH,NG)
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.727859
RSquare Adj	0.692363
Root Mean Square Error	0.021472
Mean of Response	0.148222
Observations (or Sum Wgts)	27

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	0.02836226	0.009454	20.5051
Error	23	0.01060441	0.000461	**Prob > F**
C. Total	26	0.03896667		<.0001*

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	7	0.00625958	0.000894	3.2930
Pure Error	16	0.00434483	0.000272	**Prob > F**
Total Error	23	0.01060441		0.0229*
				Max RSq

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.6565043	0.088111	7.45	<.0001*
Acid Stoichiometry	-0.295776	0.041125	-7.19	<.0001*
Mn in Feed (before trims) wt % CS	-0.031483	0.009616	-3.27	0.0033*
Hg in Feed wt % TS	0.0256121	0.011214	2.28	0.0319*

Residual by Predicted Plot

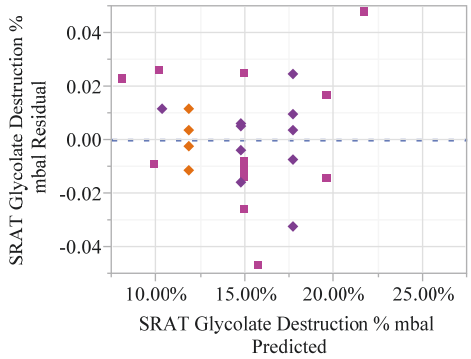
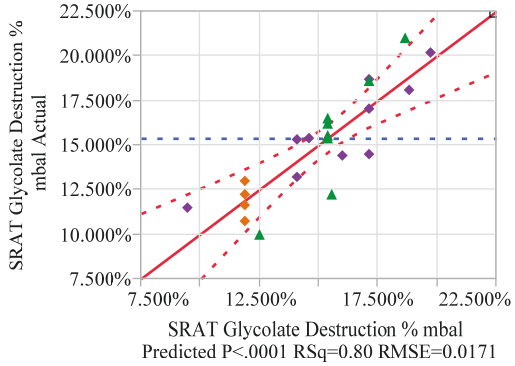


Exhibit B: Response SRAT Glycolate Destruction % mbal JMP Grouping=23 (SR+BH,43-50,57)
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.800075
RSquare Adj	0.741273
Root Mean Square Error	0.017147
Mean of Response	0.153739
Observations (or Sum Wgts)	23

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	5	0.02000381	0.004001	13.6063
Error	17	0.00499862	0.000294	**Prob > F**
C. Total	22	0.02500243		<.0001*

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	8	0.00351670	0.000440	2.6697
Pure Error	9	0.00148192	0.000165	**Prob > F**
Total Error	17	0.00499862		0.0826
				Max RSq

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.7666394	0.150469	5.09	<.0001*
Acid Stoichiometry	-0.307406	0.057189	-5.38	<.0001*
HSV	-0.041959	0.020261	-2.07	0.0539
Nitrate in Feed (before trims) mg/kg	2.6119e-5	5.091e-6	5.13	<.0001*
Mn in Feed (before trims) wt % CS	-0.065924	0.022086	-2.98	0.0083*
Hg in Feed wt % TS	0.0194065	0.00949	2.05	0.0566

Residual by Predicted Plot

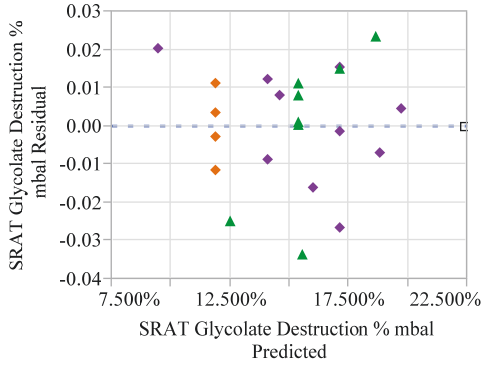
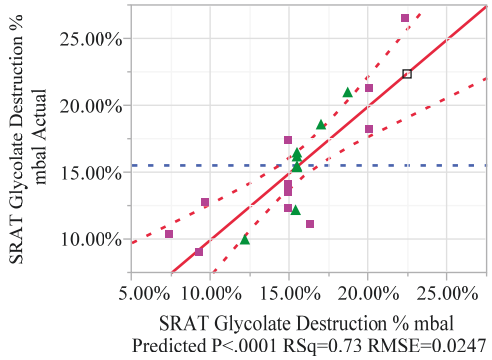


Exhibit C: Response SRAT Glycolate Destruction % mbal JMP Grouping=22 (NG,43-50,57)
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.732961
RSquare Adj	0.688454
Root Mean Square Error	0.024718
Mean of Response	0.155545
Observations (or Sum Wgts)	22

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	0.03018586	0.010062	16.4686
Error	18	0.01099760	0.000611	**Prob > F**
C. Total	21	0.04118345		<.0001*

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	9	0.00897626	0.000997	4.4408
Pure Error	9	0.00202133	0.000225	**Prob > F**
Total Error	18	0.01099760		0.0184*
				Max RSq

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.3570327	0.055823	6.40	<.0001*
Acid Stoichiometry	-0.32585	0.050862	-6.41	<.0001*
HSV	-0.052893	0.023476	-2.25	0.0370*
Nitrate in Feed (before trims) mg/kg	0.0000265	6.207e-6	4.27	0.0005*

Residual by Predicted Plot

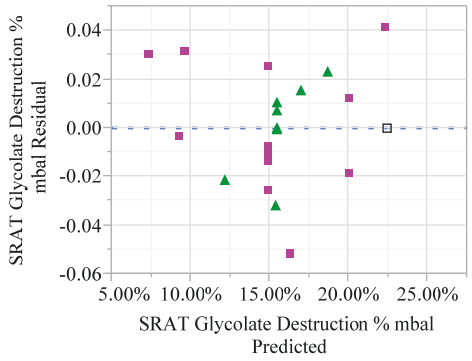
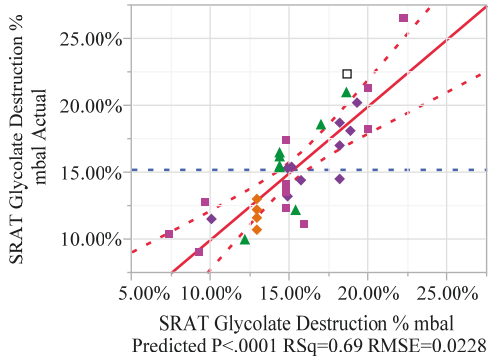


Exhibit G: Response SRAT Glycolate Destruction % mbal JMP Grouping=36 (SR+BH,NG,43-50,57)
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.694434
RSquare Adj	0.655006
Root Mean Square Error	0.022844
Mean of Response	0.152222
Observations (or Sum Wgts)	36

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	4	0.03676350	0.009191	17.6128
Error	31	0.01617672	0.000522	**Prob > F**
C. Total	35	0.05294022		<.0001*

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	16	0.01275947	0.000797	3.5005
Pure Error	15	0.00341725	0.000228	**Prob > F**
Total Error	31	0.01617672		0.0098*
				Max RSq

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.3306335	0.061483	5.38	<.0001*
Acid Stoichiometry	-0.324498	0.040304	-8.05	<.0001*
HSV	-0.017756	0.022049	-0.81	0.4268
Nitrate in Feed (before trims) mg/kg	1.9382e-5	3.973e-6	4.88	<.0001*
Hg in Feed wt % TS	0.0170886	0.011609	1.47	0.1511

Residual by Predicted Plot

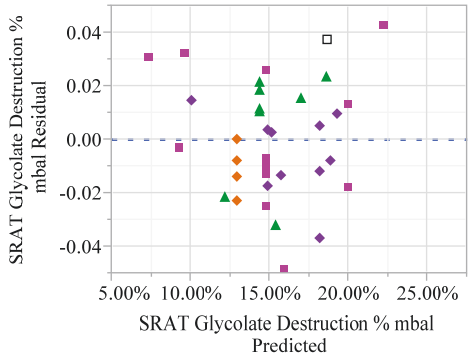
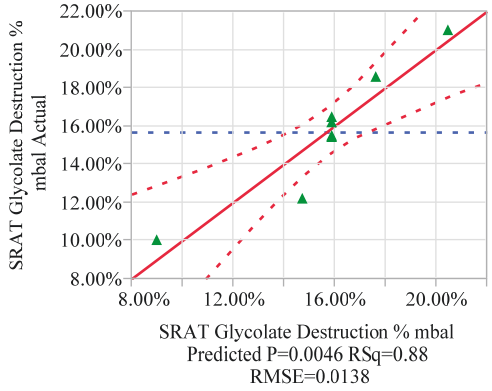


Exhibit D: Response SRAT Glycolate Destruction % mbal Group=43-50
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.883755
RSquare Adj	0.837257
Root Mean Square Error	0.013829
Mean of Response	0.15675
Observations (or Sum Wgts)	8

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	2	0.00726933	0.003635	19.0063
Error	5	0.00095617	0.000191	**Prob > F**
C. Total	7	0.00822550		0.0046*

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	2	0.00087017	0.000435	15.1774
Pure Error	3	0.00008600	0.000029	**Prob > F**
Total Error	5	0.00095617		0.0270*
				Max RSq

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.861692	0.117665	7.32	0.0007*
Acid Stoichiometry	-0.574857	0.093499	-6.15	0.0017*
HSV	-0.125946	0.035488	-3.55	0.0164*

Residual by Predicted Plot

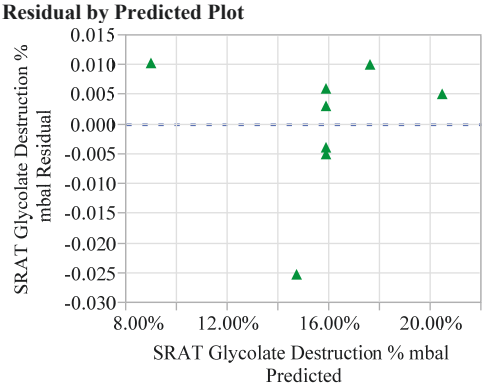
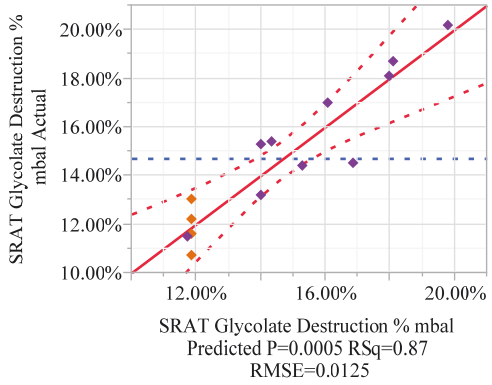


Exhibit E: Response SRAT Glycolate Destruction % mbal Group=SR+BH
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.873085
RSquare Adj	0.816678
Root Mean Square Error	0.012529
Mean of Response	0.147
Observations (or Sum Wgts)	14

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	4	0.00971918	0.002430	15.4784
Error	9	0.00141282	0.000157	**Prob > F**
C. Total	13	0.01113200		0.0005*

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	5	0.00090957	0.000182	1.4459
Pure Error	4	0.00050325	0.000126	**Prob > F**
Total Error	9	0.00141282		0.3713
				Max RSq

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.1797905	0.13426	1.34	0.2134
Acid Stoichiometry	-0.211485	0.054699	-3.87	0.0038*
HSV	-0.027713	0.018657	-1.49	0.1716
Percent Reducing Acid	0.3227978	0.183168	1.76	0.1119
Hg in Feed wt % TS	0.0245297	0.007107	3.45	0.0073*

Residual by Predicted Plot

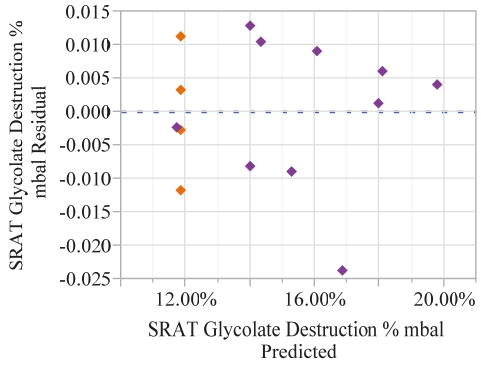
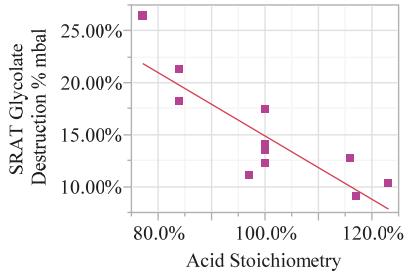
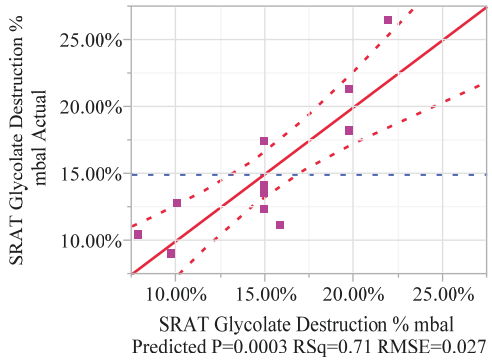


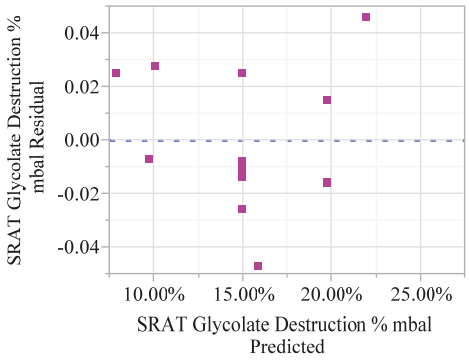
Exhibit F: Response SRAT Glycolate Destruction % mbal Group=SB9-NG
Whole Model
Regression Plot



Actual by Predicted Plot



Residual by Predicted Plot



Summary of Fit

RSquare	0.711224
RSquare Adj	0.684971
Root Mean Square Error	0.027011
Mean of Response	0.149538
Observations (or Sum Wgts)	13

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	1	0.01976578	0.019766	27.0918
Error	11	0.00802545	0.000730	**Prob > F**
C. Total	12	0.02779123		0.0003*

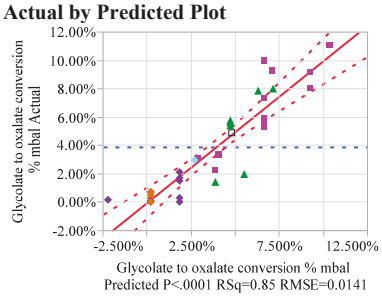
Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	5	0.00609011	0.001218	3.7762
Pure Error	6	0.00193533	0.000323	**Prob > F**
Total Error	11	0.00802545		0.0683
				Max RSq

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.4541473	0.059	7.70	<.0001*
Acid Stoichiometry	-0.305078	0.058613	-5.20	0.0003*

Exhibit X1: Response Glycolate to oxalate conversion % mbal JMP Grouping=37S *(SR+BH,NG,43-50,57,SC-18)
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.845419
RSquare Adj	0.826096
Root Mean Square Error	0.014057
Mean of Response	0.038973
Observations (or Sum Wgts)	37

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	4	0.03458184	0.008645	43.7528
Error	32	0.00632313	0.000198	**Prob > F**
C. Total	36	0.04090497		<.0001*

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	12	0.00424562	0.000354	3.4060
Pure Error	20	0.00207751	0.000104	**Prob > F**
Total Error	32	0.00632313		0.0076*
				Max RSq

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.4973771	0.040523	12.27	<.0001*
Acid Stoichiometry	-0.16207	0.022878	-7.08	<.0001*
Nitrite in Feed (before trims) mg/kg	-1.94e-5	2.979e-6	-6.51	<.0001*
Nitrate in Feed (before trims) mg/kg	-7.544e-6	2.621e-6	-2.88	0.0071*
Oxalate in Feed (before trims) mg/kg	-7.965e-6	2.563e-6	-3.11	0.0039*

Residual by Predicted Plot

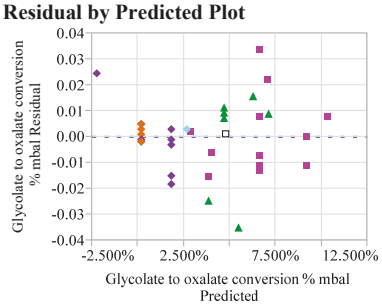
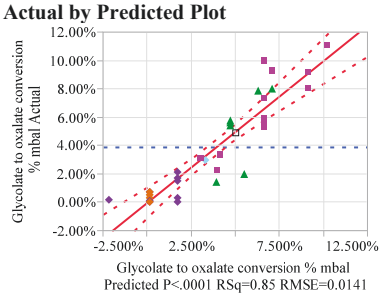


Exhibit X2: Response Glycolate to oxalate conversion % mbal JMP Grouping=37S *(SR+BH,NG,43-50,57,SC-18)
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.845407
RSquare Adj	0.826083
Root Mean Square Error	0.014057
Mean of Response	0.038973
Observations (or Sum Wgts)	37

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	4	0.03458136	0.008645	43.7489
Error	32	0.00632361	0.000198	**Prob > F**
C. Total	36	0.04090497		<.0001*

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	12	0.00424611	0.000354	3.4064
Pure Error	20	0.00207751	0.000104	**Prob > F**
Total Error	32	0.00632361		0.0076*
				Max RSq

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.2165446	0.027064	8.00	<.0001*
Acid Stoichiometry	-0.155885	0.022774	-6.84	<.0001*
Nitrate in Feed (before trims) mg/kg	7.1486e-6	4.116e-6	1.74	0.0921
Oxalate in Feed (before trims) mg/kg	-2.474e-5	4.385e-6	-5.64	<.0001*
Runitrosyl (Y/N)[N]	-0.051633	0.00793	-6.51	<.0001*

Residual by Predicted Plot

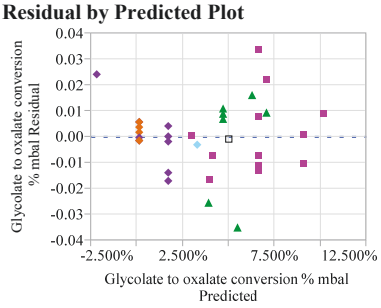
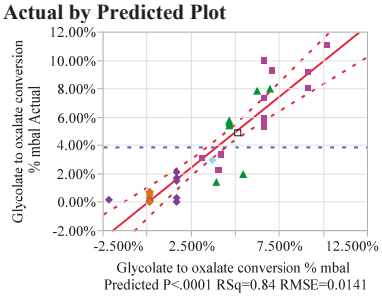


Exhibit X3: Response Glycolate to oxalate conversion % mbal JMP Grouping=37S *(SR+BH,NG,43-50,57,SC-18)
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.844098
RSquare Adj	0.824611
Root Mean Square Error	0.014117
Mean of Response	0.038973
Observations (or Sum Wgts)	37

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	4	0.03452782	0.008632	43.3144
Error	32	0.00637716	0.000199	**Prob > F**
C. Total	36	0.04090497		<.0001*

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	12	0.00429965	0.000358	3.4494
Pure Error	20	0.00207751	0.000104	**Prob > F**
Total Error	32	0.00637716		0.0071*
				Max RSq

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.3878065	0.100947	3.84	0.0005*
Acid Stoichiometry	-0.15326	0.023565	-6.50	<.0001*
Nitrite in Feed (before trims) mg/kg	-0.000016	4.223e-6	-3.79	0.0006*
Mn in Feed (before trims) wt% CS	0.0131395	0.008726	1.51	0.1419
Fe in Feed (before trims) wt% CS	-0.004878	0.001424	-3.42	0.0017*

Residual by Predicted Plot

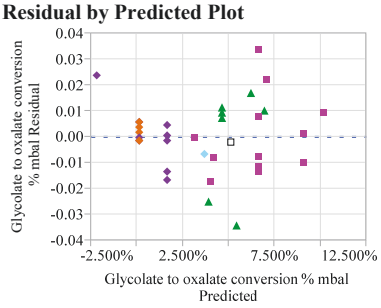
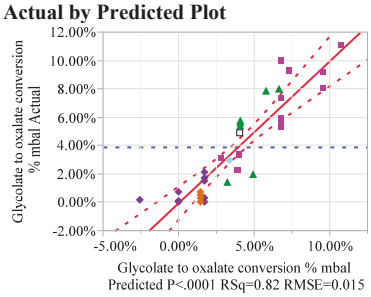


Exhibit X4: Response Glycolate to oxalate conversion % mbal JMP Grouping=37S *(SR+BH,NG,43-50,57,SC-18)
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.823914
RSquare Adj	0.801903
Root Mean Square Error	0.015003
Mean of Response	0.038973
Observations (or Sum Wgts)	37

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	4	0.03370217	0.008426	37.4323
Error	32	0.00720281	0.000225	**Prob > F**
C. Total	36	0.04090497		<.0001*

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	13	0.00513142	0.000395	3.6207
Pure Error	19	0.00207138	0.000109	**Prob > F**
Total Error	32	0.00720281		0.0056*
				Max RSq

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.4394072	0.036227	12.13	<.0001*
Acid Stoichiometry	-0.171975	0.02432	-7.07	<.0001*
Nitrite in Feed (before trims) mg/kg	-1.663e-5	2.91e-6	-5.71	<.0001*
Nitrate in Feed (before trims) mg/kg	-6.55e-6	2.755e-6	-2.38	0.0236*
Rh wt%	0.4763769	0.222792	2.14	0.0402*

Residual by Predicted Plot

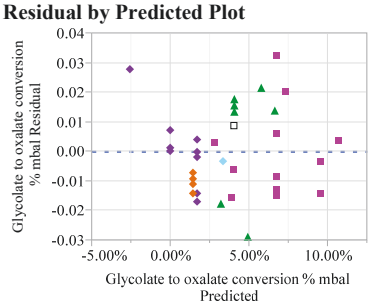
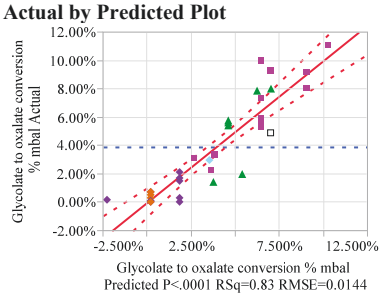


Exhibit X5: Response Glycolate to oxalate conversion % mbal JMP Grouping=37S *(SR+BH,NG,43-50,57,SC-18)
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.833052
RSquare Adj	0.817875
Root Mean Square Error	0.014385
Mean of Response	0.038973
Observations (or Sum Wgts)	37

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	0.03407597	0.011359	54.8888
Error	33	0.00682900	0.000207	**Prob > F**
C. Total	36	0.04090497		<.0001*

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	13	0.00475149	0.000365	3.5186
Pure Error	20	0.00207751	0.000104	**Prob > F**
Total Error	33	0.00682900		0.0058*
				Max RSq

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.5273581	0.040772	12.93	<.0001*
Acid Stoichiometry	-0.164533	0.022768	-7.23	<.0001*
Nitrite in Feed (before trims) mg/kg	-2.139e-5	2.303e-6	-9.29	<.0001*
Fe in Feed (before trims) wt% CS	-0.003268	0.000959	-3.41	0.0017*

Residual by Predicted Plot

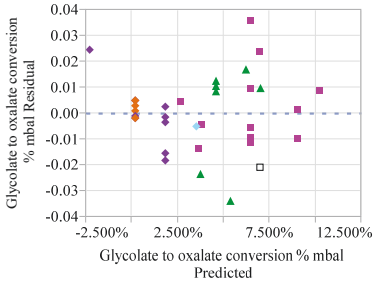
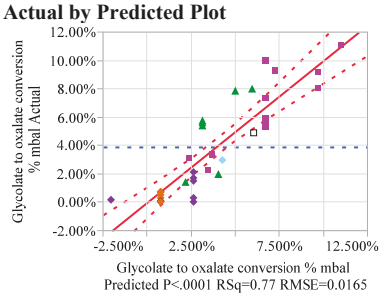


Exhibit X6: Response Glycolate to oxalate conversion % mbal JMP Grouping=37S *(SR+BH,NG,43-50,57,SC-18)
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.77429
RSquare Adj	0.761013
Root Mean Square Error	0.016479
Mean of Response	0.038973
Observations (or Sum Wgts)	37

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	2	0.03167232	0.015836	58.3180
Error	34	0.00923265	0.000272	**Prob > F**
C. Total	36	0.04090497		<.0001*

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	14	0.00715514	0.000511	4.9201
Pure Error	20	0.00207751	0.000104	**Prob > F**
Total Error	34	0.00923265		0.0007*
				Max RSq

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.4459707	0.037856	11.78	<.0001*
Acid Stoichiometry	-0.187849	0.024877	-7.55	<.0001*
Nitrite in Feed (before trims) mg/kg	-1.875e-5	2.485e-6	-7.55	<.0001*

Residual by Predicted Plot

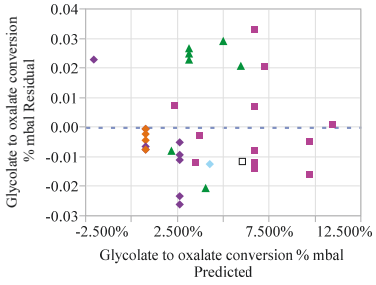
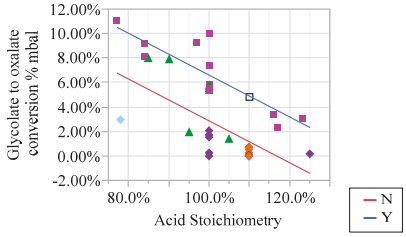
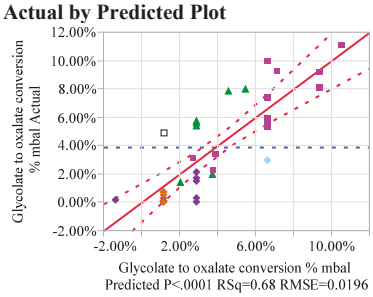


Exhibit X7: Response Glycolate to oxalate conversion % mbal JMP Grouping=37S *(SR+BH,NG,43-50,57,SC-18)
Whole Model
Regression Plot



Actual by Predicted Plot



Summary of Fit

RSquare	0.679509
RSquare Adj	0.660657
Root Mean Square Error	0.019636
Mean of Response	0.038973
Observations (or Sum Wgts)	37

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	2	0.02779532	0.013898	36.0437
Error	34	0.01310966	0.000386	**Prob > F**
C. Total	36	0.04090497		<.0001*

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	12	0.00468093	0.000390	1.0182
Pure Error	22	0.00842872	0.000383	**Prob > F**
Total Error	34	0.01310966		0.4661
				Max RSq

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.2182302	0.030481	7.16	<.0001*
Acid Stoichiometry	-0.170654	0.029893	-5.71	<.0001*
Runitrosyl (Y/N)[N]	-0.0187	0.00341	-5.48	<.0001*

Residual by Predicted Plot

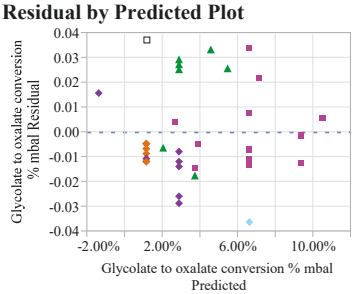
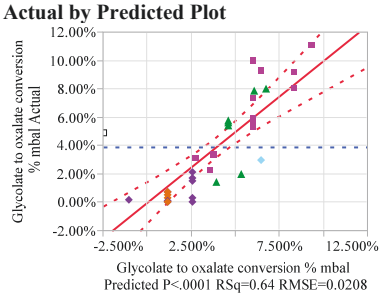


Exhibit X8: Response Glycolate to oxalate conversion % mbal JMP Grouping=37S *(SR+BH,NG,43-50,57,SC-18)
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.640494
RSquare Adj	0.619347
Root Mean Square Error	0.020797
Mean of Response	0.038973
Observations (or Sum Wgts)	37

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	2	0.02619941	0.013100	30.2872
Error	34	0.01470556	0.000433	**Prob > F**
C. Total	36	0.04090497		<.0001*

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	14	0.01262806	0.000902	8.6835
Pure Error	20	0.00207751	0.000104	**Prob > F**
Total Error	34	0.01470556		<.0001*
				Max RSq

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.28688	0.033926	8.46	<.0001*
Acid Stoichiometry	-0.143295	0.032989	-4.34	0.0001*
Nitrate in Feed (before trims) mg/kg	-1.454e-5	3.025e-6	-4.81	<.0001*

Residual by Predicted Plot

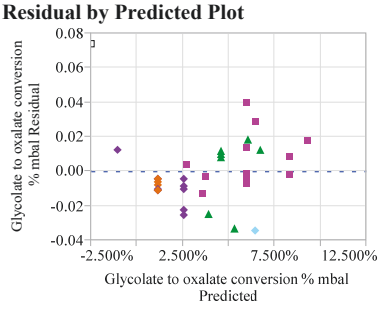
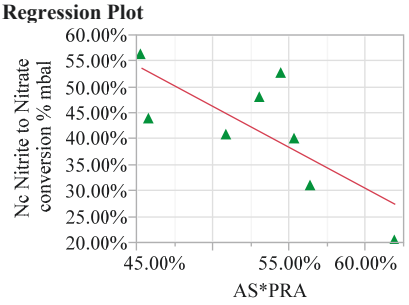
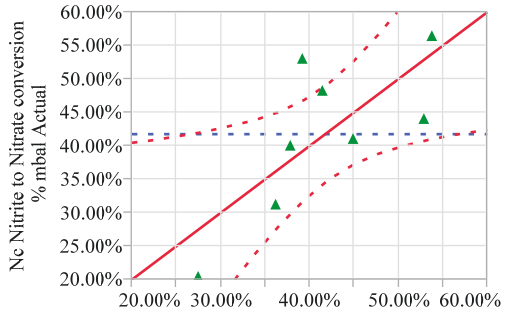


Exhibit n1a: Response Nc Nitrite to Nitrate conversion % mbal Group=43-50
Whole Model
Regression Plot



Actual by Predicted Plot



Summary of Fit

RSquare	0.564151
RSquare Adj	0.491509
Root Mean Square Error	0.083171
Mean of Response	0.417625
Observations (or Sum Wgts)	8

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	1	0.05372174	0.053722	7.7662
Error	6	0.04150413	0.006917	**Prob > F**
C. Total	7	0.09522588		0.0317*

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	1.25432	0.301672	4.16	0.0060*
AS*PRA	-1.582391	0.567817	-2.79	0.0317*

Residual by Predicted Plot

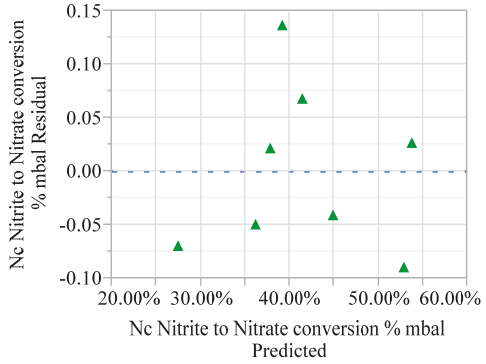
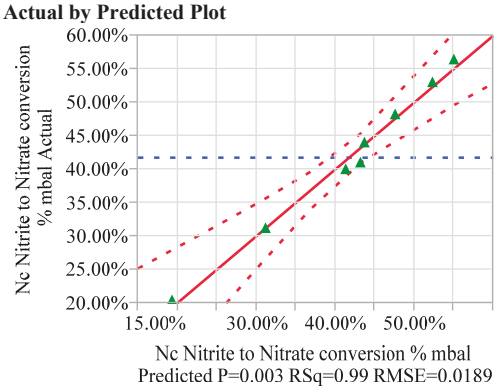


Exhibit n1b: Response Nc Nitrite to Nitrate conversion % mbal Group=43-50
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.98874
RSquare Adj	0.973727
Root Mean Square Error	0.018905
Mean of Response	0.417625
Observations (or Sum Wgts)	8

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	4	0.09415363	0.023538	65.8575
Error	3	0.00107224	0.000357	**Prob > F**
C. Total	7	0.09522588		0.0030*

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	-14.24402	2.798725	-5.09	0.0147*
Acid Stoichiometry	15.509461	2.852279	5.44	0.0122*
Percent Reducing Acid	26.499563	5.23474	5.06	0.0149*
AS*PRA	-28.6489	5.282494	-5.42	0.0123*
HSV	0.2552771	0.057101	4.47	0.0209*

Residual by Predicted Plot

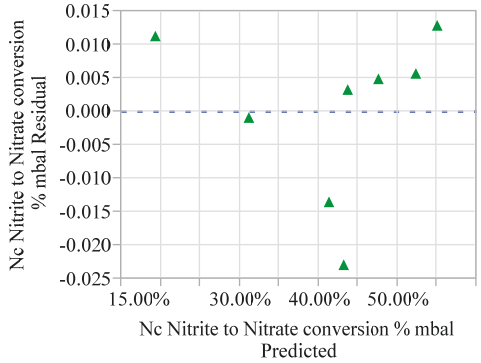
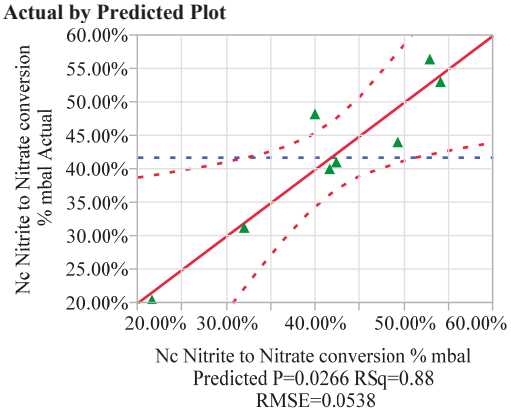


Exhibit n1c: Response Nc Nitrite to Nitrate conversion % mbal Group=43-50
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.878344
RSquare Adj	0.787101
Root Mean Square Error	0.053816
Mean of Response	0.417625
Observations (or Sum Wgts)	8

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	0.08364104	0.027880	9.6265
Error	4	0.01158483	0.002896	**Prob > F**
C. Total	7	0.09522588		0.0266*

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.901005	0.528864	1.70	0.1637
Acid Stoichiometry	0.056547	0.369612	0.15	0.8858
Percent Reducing Acid	-1.880192	0.398507	-4.72	0.0092*
HSV	0.4164837	0.138783	3.00	0.0399*

Residual by Predicted Plot

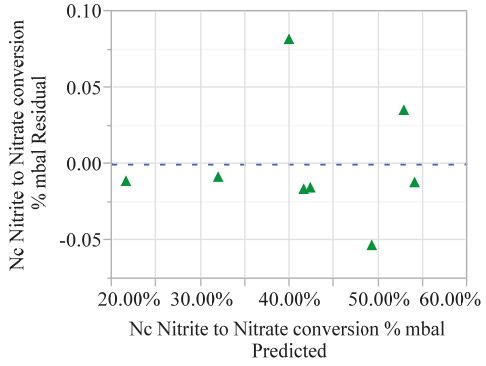
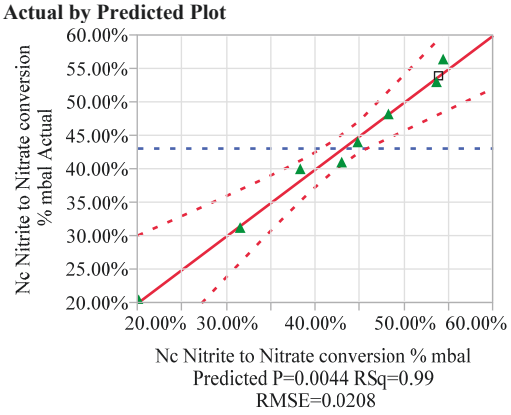


Exhibit n2a: Response Nc Nitrite to Nitrate conversion % mbal
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.987985
RSquare Adj	0.967959
Root Mean Square Error	0.020829
Mean of Response	0.431111
Observations (or Sum Wgts)	9

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	5	0.10701936	0.021404	49.3357
Error	3	0.00130153	0.000434	**Prob > F**
C. Total	8	0.10832089		0.0044*

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	-8.578311	1.944634	-4.41	0.0216*
Acid Stoichiometry	0.2431742	0.1481	1.64	0.1991
Percent Reducing Acid	-2.059447	0.15857	-12.99	0.0010*
SASV	125.90107	25.86001	4.87	0.0166*
HSV	-46.6992	9.677684	-4.83	0.0170*
Oxalate in Feed (before trims) mg/kg	-0.000516	0.000155	-3.32	0.0450*

Residual by Predicted Plot

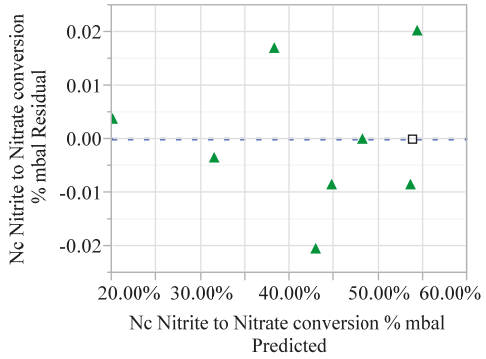
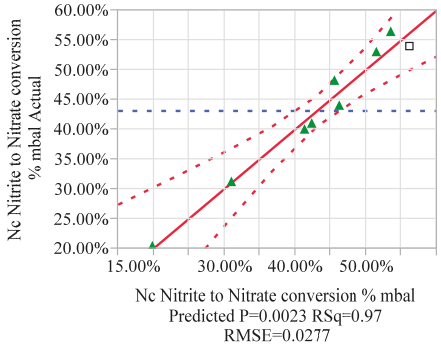


Exhibit n2b: Response Nc Nitrite to Nitrate conversion % mbal
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.971752
RSquare Adj	0.943505
Root Mean Square Error	0.027658
Mean of Response	0.431111
Observations (or Sum Wgts)	9

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	4	0.10526108	0.026315	34.4012
Error	4	0.00305981	0.000765	**Prob > F**
C. Total	8	0.10832089		0.0023*

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	-9.276911	2.696041	-3.44	0.0263*
Acid Stoichiometry	10.425361	2.731994	3.82	0.0188*
Percent Reducing Acid	17.387915	5.166749	3.37	0.0282*
AS*PRA	-19.41222	5.185294	-3.74	0.0201*
HSV	0.2994376	0.078915	3.79	0.0192*

Residual by Predicted Plot

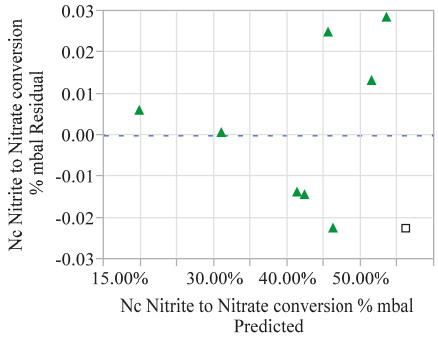
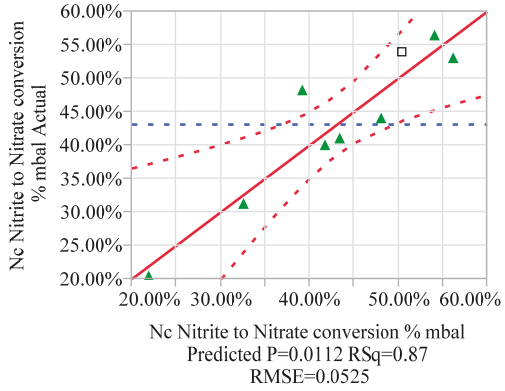


Exhibit n2c: Response Nc Nitrite to Nitrate conversion % mbal
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.872777
RSquare Adj	0.796444
Root Mean Square Error	0.052499
Mean of Response	0.431111
Observations (or Sum Wgts)	9

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	0.09454002	0.031513	11.4337
Error	5	0.01378087	0.002756	**Prob > F**
C. Total	8	0.10832089		0.0112*

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.7690814	0.494298	1.56	0.1805
Acid Stoichiometry	0.216233	0.313056	0.69	0.5205
Percent Reducing Acid	-1.940125	0.382911	-5.07	0.0039*
HSV	0.4287252	0.13469	3.18	0.0245*

Residual by Predicted Plot

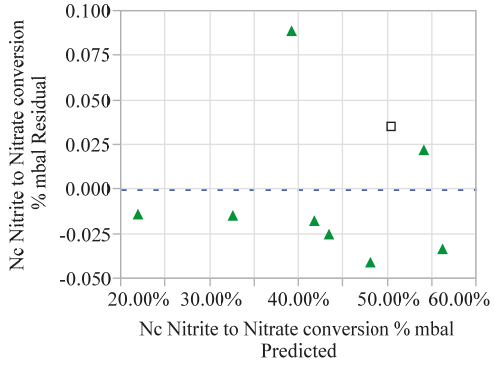
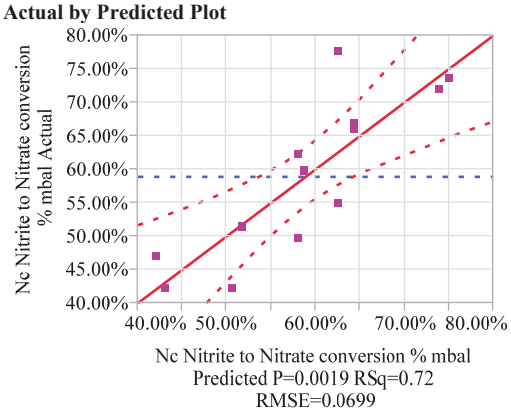


Exhibit n3a: Response Nc Nitrite to Nitrate conversion % mbal Group=SB9-NG
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.715372
RSquare Adj	0.658446
Root Mean Square Error	0.06986
Mean of Response	0.588769
Observations (or Sum Wgts)	13

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	2	0.12266077	0.061330	12.5668
Error	10	0.04880354	0.004880	**Prob > F**
C. Total	12	0.17146431		0.0019*

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	-0.113419	0.152631	-0.74	0.4745
Acid Stoichiometry	1.3087671	0.341027	3.84	0.0033*
AS*PRA	-1.053593	0.525796	-2.00	0.0729

Residual by Predicted Plot

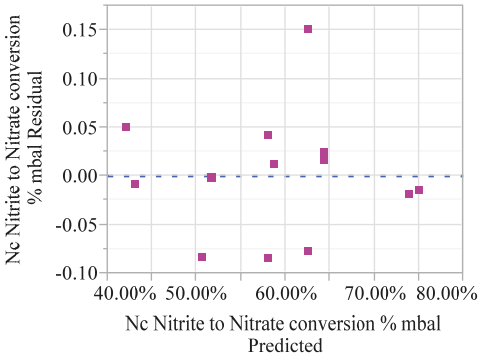
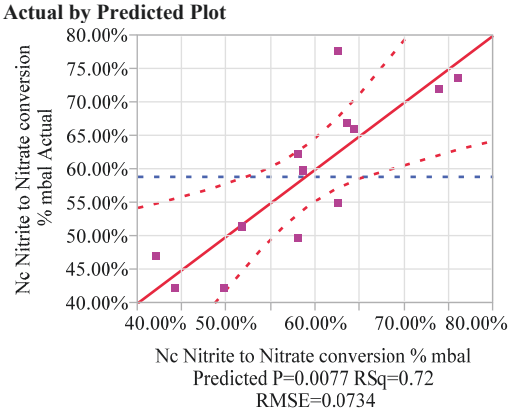


Exhibit n3b: Response Nc Nitrite to Nitrate conversion % mbal Group=SB9-NG
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.717365
RSquare Adj	0.623154
Root Mean Square Error	0.07338
Mean of Response	0.588769
Observations (or Sum Wgts)	13

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	0.12300257	0.041001	7.6144
Error	9	0.04846173	0.005385	**Prob > F**
C. Total	12	0.17146431		0.0077*

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	-0.908918	3.161453	-0.29	0.7802
Acid Stoichiometry	2.0982718	3.154002	0.67	0.5226
Percent Reducing Acid	1.3655949	5.420133	0.25	0.8067
AS*PRA	-2.40879	5.407145	-0.45	0.6665

Residual by Predicted Plot

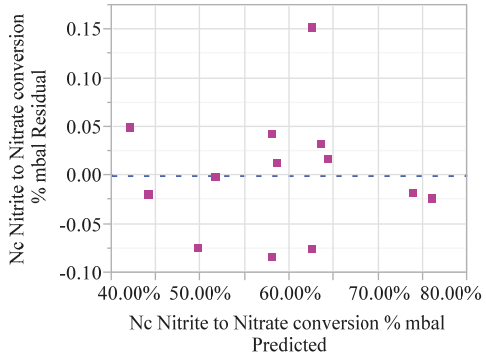
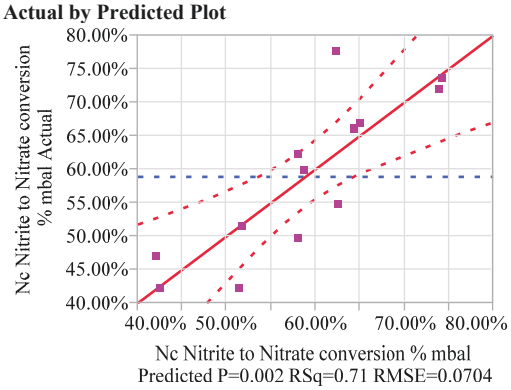


Exhibit n3c: Response Nc Nitrite to Nitrate conversion % mbal Group=SB9-NG
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.711133
RSquare Adj	0.65336
Root Mean Square Error	0.070378
Mean of Response	0.588769
Observations (or Sum Wgts)	13

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	2	0.12193396	0.060967	12.3090
Error	10	0.04953034	0.004953	**Prob > F**
C. Total	12	0.17146431		0.0020*

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.4904446	0.34244	1.43	0.1826
Acid Stoichiometry	0.6950097	0.15272	4.55	0.0011*
Percent Reducing Acid	-1.036353	0.530969	-1.95	0.0795

Residual by Predicted Plot

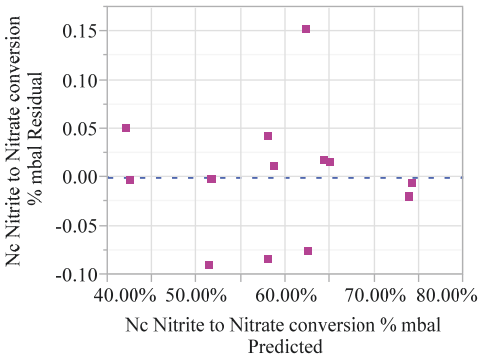
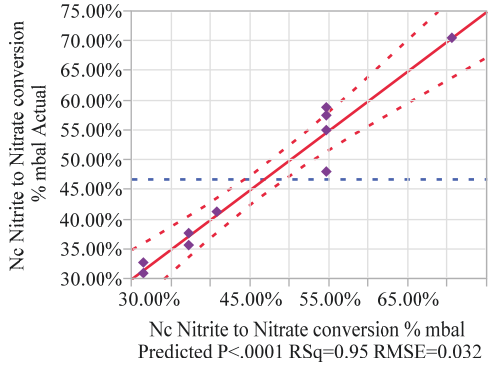


Exhibit n4a: Response Nc Nitrite to Nitrate conversion % mbal Data Set=SR
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.954371
RSquare Adj	0.941334
Root Mean Square Error	0.032029
Mean of Response	0.4679
Observations (or Sum Wgts)	10

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	2	0.15019206	0.075096	73.2049
Error	7	0.00718084	0.001026	**Prob > F**
C. Total	9	0.15737290		<.0001*

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	2	0.00011034	0.000055	0.0390
Pure Error	5	0.00707050	0.001414	**Prob > F**
Total Error	7	0.00718084		0.9620
				Max RSq

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	-0.998951	0.145946	-6.84	0.0002*
Acid Stoichiometry	2.1828893	0.23612	9.24	<.0001*
AS*PRA	-1.488804	0.439065	-3.39	0.0116*

Residual by Predicted Plot

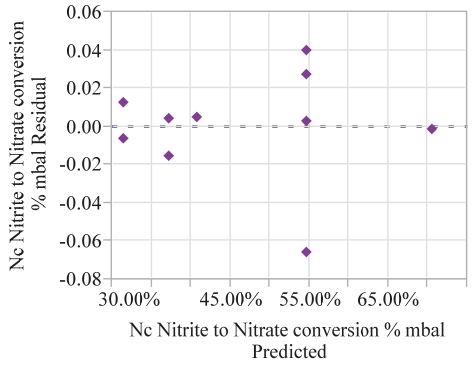
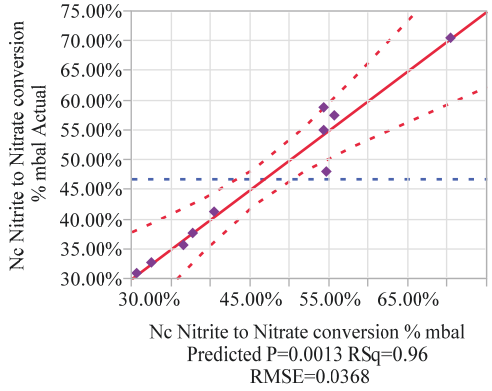


Exhibit n4b: Response Nc Nitrite to Nitrate conversion % mbal Data Set=SR
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.956907
RSquare Adj	0.922433
Root Mean Square Error	0.036828
Mean of Response	0.4679
Observations (or Sum Wgts)	10

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	4	0.15059123	0.037648	27.7571
Error	5	0.00678167	0.001356	**Prob > F**
C. Total	9	0.15737290		0.0013*

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	4	0.00609717	0.001524	2.2269
Pure Error	1	0.00068450	0.000685	**Prob > F**
Total Error	5	0.00678167		0.4605
				Max RSq

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	-0.49088	7.08295	-0.07	0.9474
Acid Stoichiometry	1.7532779	6.786755	0.26	0.8064
Percent Reducing Acid	-0.91302	12.91917	-0.07	0.9464
AS*PRA	-0.673243	12.42083	-0.05	0.9589
HSV	-0.030541	0.056955	-0.54	0.6148

Residual by Predicted Plot

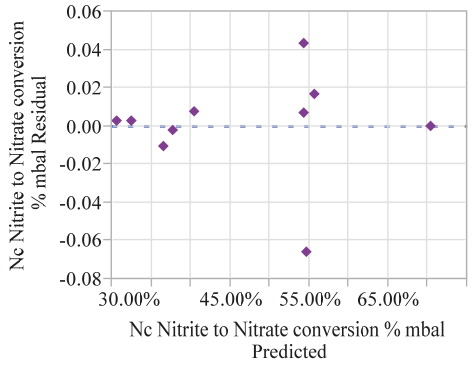
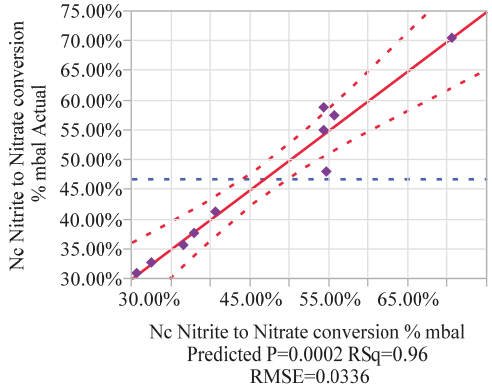


Exhibit n4c: Response Nc Nitrite to Nitrate conversion % mbal
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.956882
RSquare Adj	0.935323
Root Mean Square Error	0.033629
Mean of Response	0.4679
Observations (or Sum Wgts)	10

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	0.15058725	0.050196	44.3840
Error	6	0.00678565	0.001131	**Prob > F**
C. Total	9	0.15737290		0.0002*

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	5	0.00610115	0.001220	1.7827
Pure Error	1	0.00068450	0.000685	**Prob > F**
Total Error	6	0.00678565		0.5124
				Max RSq

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	-0.107524	0.34893	-0.31	0.7684
Acid Stoichiometry	1.3855204	0.146817	9.44	<.0001*
Percent Reducing Acid	-1.612666	0.491641	-3.28	0.0168*
HSV	-0.031374	0.050077	-0.63	0.5540

Residual by Predicted Plot

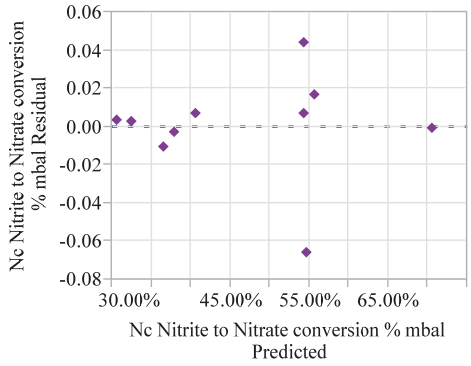
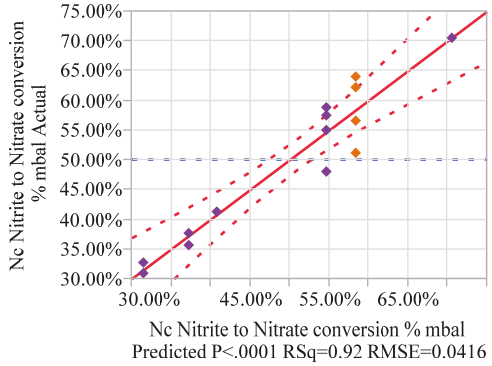


Exhibit n5a: Response Nc Nitrite to Nitrate conversion % mbal Group=SR+BH
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.915913
RSquare Adj	0.890687
Root Mean Square Error	0.041641
Mean of Response	0.501143
Observations (or Sum Wgts)	14

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	0.18887013	0.062957	36.3081
Error	10	0.01733959	0.001734	**Prob > F**
C. Total	13	0.20620971		<.0001*

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	2	0.00011034	0.000055	0.0256
Pure Error	8	0.01722925	0.002154	**Prob > F**
Total Error	10	0.01733959		0.9748
				Max RSq

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	-0.867286	0.205482	-4.22	0.0018*
Acid Stoichiometry	2.1828893	0.306983	7.11	<.0001*
AS*PRA	-1.488804	0.570834	-2.61	0.0261*
Hg in Feed wt % TS	-0.061526	0.022347	-2.75	0.0204*

Residual by Predicted Plot

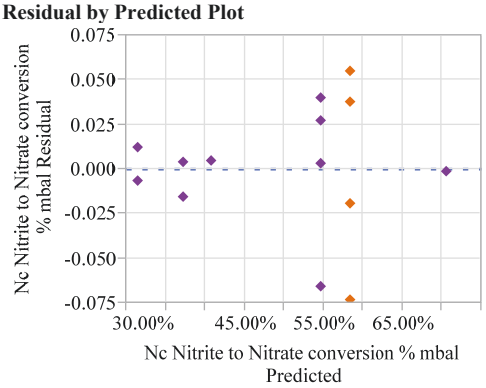
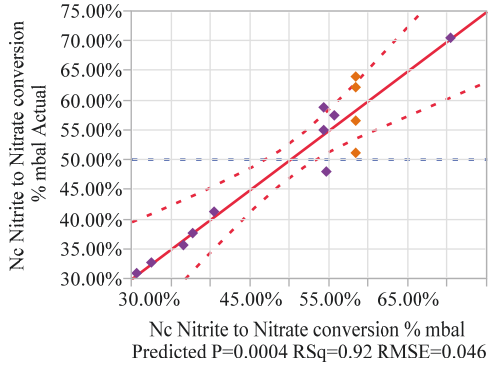


Exhibit n5b: Response Nc Nitrite to Nitrate conversion % mbal Group=SR+BH
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.917849
RSquare Adj	0.866504
Root Mean Square Error	0.046017
Mean of Response	0.501143
Observations (or Sum Wgts)	14

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	5	0.18926930	0.037854	17.8762
Error	8	0.01694042	0.002118	**Prob > F**
C. Total	13	0.20620971		0.0004*

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	4	0.00609717	0.001524	0.5623
Pure Error	4	0.01084325	0.002711	**Prob > F**
Total Error	8	0.01694042		0.7046
				Max RSq

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	-0.351759	8.843496	-0.04	0.9692
Acid Stoichiometry	1.7532779	8.480004	0.21	0.8414
Percent Reducing Acid	-0.91302	16.14242	-0.06	0.9563
AS*PRA	-0.673243	15.51974	-0.04	0.9665
HSV	-0.030541	0.071165	-0.43	0.6791
Hg in Feed wt % TS	-0.06501	0.026336	-2.47	0.0388*

Residual by Predicted Plot

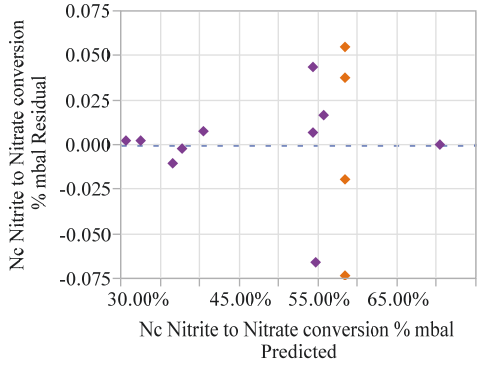
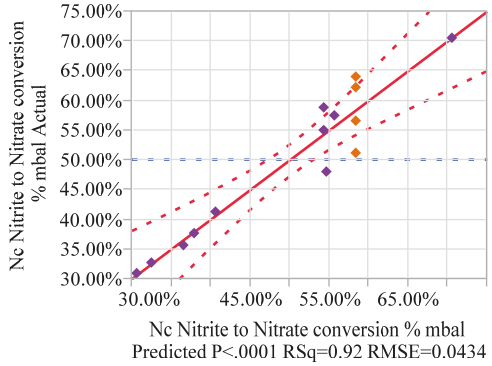


Exhibit n5c: Response Nc Nitrite to Nitrate conversion % mbal Group=SR+BH
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.917829
RSquare Adj	0.881309
Root Mean Square Error	0.04339
Mean of Response	0.501143
Observations (or Sum Wgts)	14

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	4	0.18926531	0.047316	25.1320
Error	9	0.01694440	0.001883	**Prob > F**
C. Total	13	0.20620971		<.0001*

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	5	0.00610115	0.001220	0.4501
Pure Error	4	0.01084325	0.002711	**Prob > F**
Total Error	9	0.01694440		0.7977
				Max RSq

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.0312732	0.464961	0.07	0.9478
Acid Stoichiometry	1.3855204	0.189429	7.31	<.0001*
Percent Reducing Acid	-1.612666	0.634337	-2.54	0.0316*
HSV	-0.031374	0.064612	-0.49	0.6389
Hg in Feed wt % TS	-0.064859	0.024614	-2.64	0.0271*

Residual by Predicted Plot

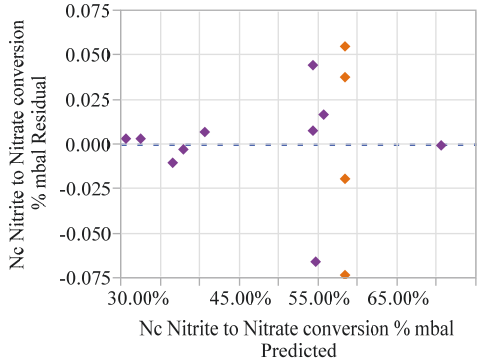
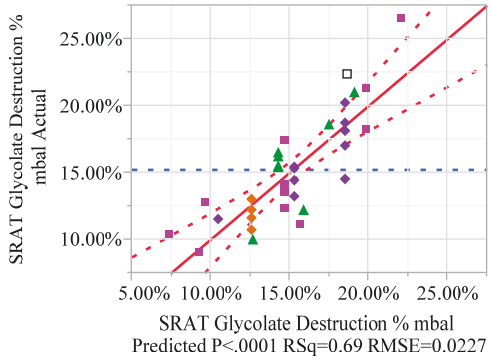


Exhibit G1: Response SRAT Glycolate Destruction % mbal JMP Grouping=36 (SR+BH,NG,43-50,57)
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.688042
RSquare Adj	0.658796
Root Mean Square Error	0.022718
Mean of Response	0.152222
Observations (or Sum Wgts)	36

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	0.03642510	0.012142	23.5260
Error	32	0.01651512	0.000516	**Prob > F**
C. Total	35	0.05294022		<.0001*

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	13	0.01208429	0.000930	3.9861
Pure Error	19	0.00443083	0.000233	**Prob > F**
Total Error	32	0.01651512		0.0033*
				Max RSq

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.2997349	0.047777	6.27	<.0001*
Acid Stoichiometry	-0.319911	0.03968	-8.06	<.0001*
Nitrate in Feed (before trims) mg/kg	1.9179e-5	3.943e-6	4.86	<.0001*
Hg in Feed wt % TS	0.0232576	0.008674	2.68	0.0115*

Residual by Predicted Plot

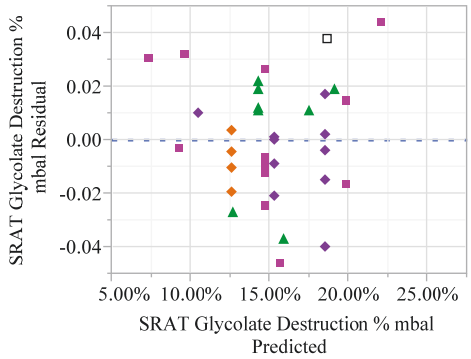
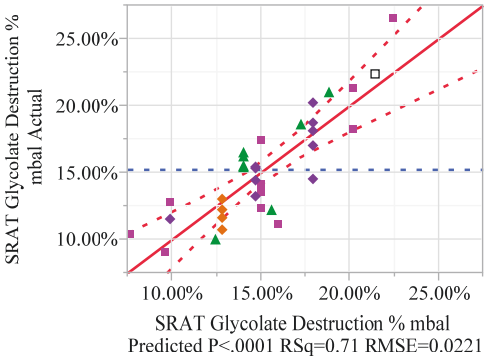


Exhibit G2: Response SRAT Glycolate Destruction % mbal JMP Grouping=36 (SR+BH,NG,43-50,57)
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.713019
RSquare Adj	0.675989
Root Mean Square Error	0.022138
Mean of Response	0.152222
Observations (or Sum Wgts)	36

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	4	0.03774737	0.009437	19.2552
Error	31	0.01519286	0.000490	**Prob > F**
C. Total	35	0.05294022		<.0001*

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	12	0.01076202	0.000897	3.8457
Pure Error	19	0.00443083	0.000233	**Prob > F**
Total Error	31	0.01519286		0.0044*
				Max RSq

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.3892385	0.071671	5.43	<.0001*
Acid Stoichiometry	-0.321662	0.038682	-8.32	<.0001*
Nitrate in Feed (before trims) mg/kg	0.0000218	4.159e-6	5.24	<.0001*
Hg in Feed wt % TS	0.0162676	0.009464	1.72	0.0956
Nitrite in Feed (before trims) mg/kg	-8.056e-6	4.905e-6	-1.64	0.1106

Residual by Predicted Plot

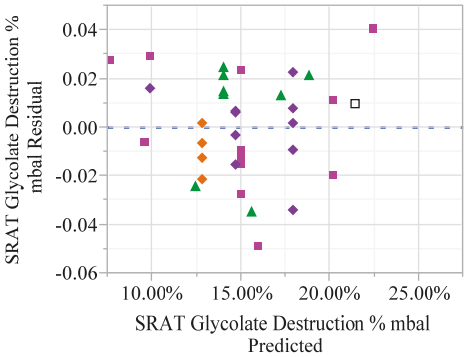
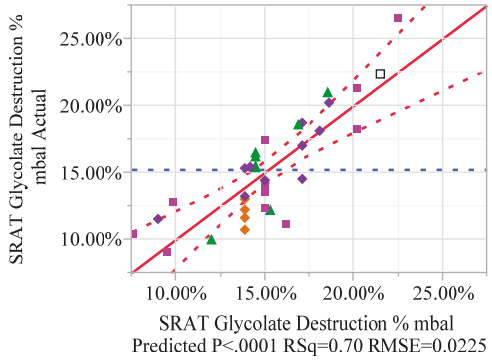


Exhibit G3: Response SRAT Glycolate Destruction % mbal JMP Grouping=36 (SR+BH,NG,43-50,57)
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.702519
RSquare Adj	0.664134
Root Mean Square Error	0.022539
Mean of Response	0.152222
Observations (or Sum Wgts)	36

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	4	0.03719152	0.009298	18.3021
Error	31	0.01574871	0.000508	**Prob > F**
C. Total	35	0.05294022		<.0001*

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	16	0.01233146	0.000771	3.3831
Pure Error	15	0.00341725	0.000228	**Prob > F**
Total Error	31	0.01574871		0.0115*
				Max RSq

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.462695	0.054177	8.54	<.0001*
Acid Stoichiometry	-0.325231	0.039756	-8.18	<.0001*
Nitrate in Feed (before trims) mg/kg	2.0649e-5	4.129e-6	5.00	<.0001*
Nitrite in Feed (before trims) mg/kg	-8.794e-6	5.02e-6	-1.75	0.0897
HSV	-0.024384	0.018398	-1.33	0.1948

Residual by Predicted Plot

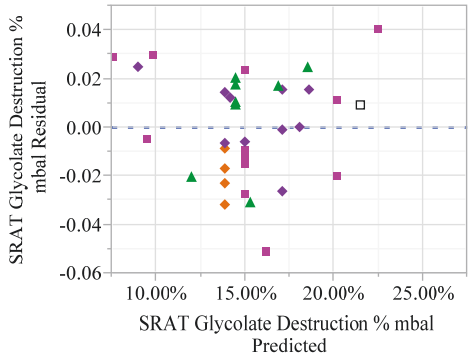
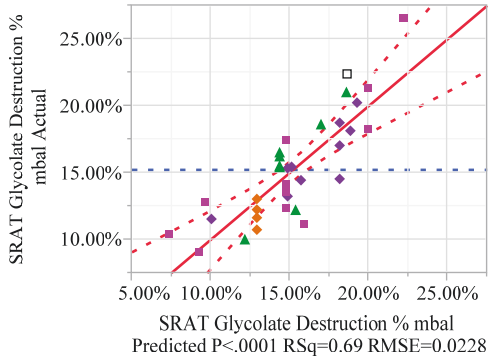


Exhibit G4: Response SRAT Glycolate Destruction % mbal JMP Grouping=36 (SR+BH,NG,43-50,57)
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.694434
RSquare Adj	0.655006
Root Mean Square Error	0.022844
Mean of Response	0.152222
Observations (or Sum Wgts)	36

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	4	0.03676350	0.009191	17.6128
Error	31	0.01617672	0.000522	**Prob > F**
C. Total	35	0.05294022		<.0001*

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	16	0.01275947	0.000797	3.5005
Pure Error	15	0.00341725	0.000228	**Prob > F**
Total Error	31	0.01617672		0.0098*
				Max RSq

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.3306335	0.061483	5.38	<.0001*
Acid Stoichiometry	-0.324498	0.040304	-8.05	<.0001*
Nitrate in Feed (before trims) mg/kg	1.9382e-5	3.973e-6	4.88	<.0001*
HSV	-0.017756	0.022049	-0.81	0.4268
Hg in Feed wt % TS	0.0170886	0.011609	1.47	0.1511

Residual by Predicted Plot

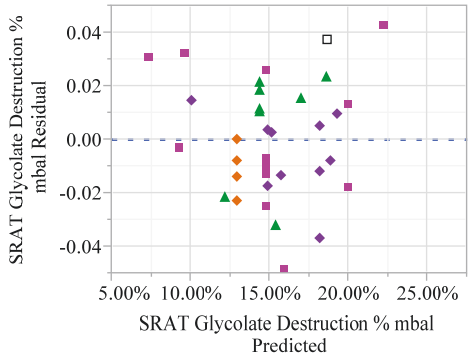
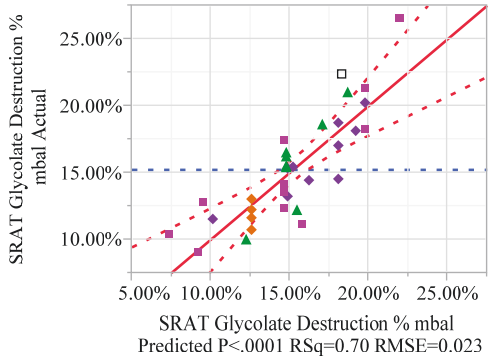


Exhibit G5: Response SRAT Glycolate Destruction % mbal JMP Grouping=36 (SR+BH,NG,43-50,57)
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.69898
RSquare Adj	0.64881
Root Mean Square Error	0.023048
Mean of Response	0.152222
Observations (or Sum Wgts)	36

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	5	0.03700418	0.007401	13.9323
Error	30	0.01593605	0.000531	**Prob > F**
C. Total	35	0.05294022		<.0001*

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	15	0.01251880	0.000835	3.6634
Pure Error	15	0.00341725	0.000228	**Prob > F**
Total Error	30	0.01593605		0.0083*
				Max RSq

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.4111826	0.13479	3.05	0.0047*
Acid Stoichiometry	-0.31898	0.041483	-7.69	<.0001*
Nitrate in Feed (before trims) mg/kg	0.0000173	5.063e-6	3.42	0.0018*
HSV	-0.027876	0.026851	-1.04	0.3075
Hg in Feed wt % TS	0.0202213	0.012603	1.60	0.1191
Mn in Feed (before trims) wt % CS	-0.008775	0.013037	-0.67	0.5060

Residual by Predicted Plot

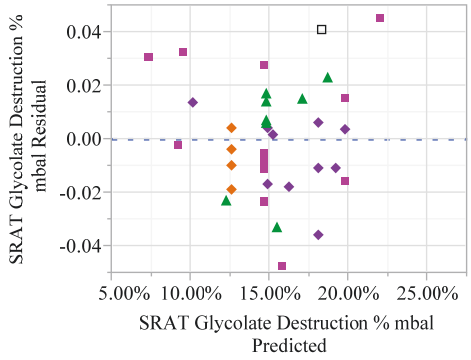
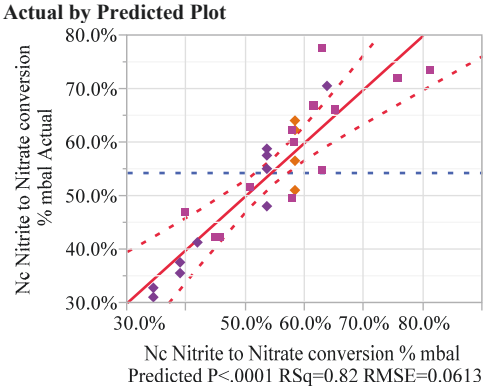


Exhibit N1a: Response Nc Nitrite to Nitrate conversion % mbal JMP Grouping=27 (SR+BH,NG)
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.815949
RSquare Adj	0.772128
Root Mean Square Error	0.061349
Mean of Response	0.543333
Observations (or Sum Wgts)	27

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	5	0.35039474	0.070079	18.6198
Error	21	0.07903726	0.003764	**Prob > F**
C. Total	26	0.42943200		<.0001*

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	13	0.06180801	0.004754	2.2076
Pure Error	8	0.01722925	0.002154	**Prob > F**
Total Error	21	0.07903726		0.1322
				Max RSq

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	-2.037228	2.181393	-0.93	0.3610
Acid Stoichiometry	4.721522	2.154244	2.19	0.0398*
Percent Reducing Acid	5.5491975	3.816971	1.45	0.1608
AS*PRA	-6.744008	3.746046	-1.80	0.0862
Nitrite in Feed (before trims) mg/kg	-0.00012	1.653e-5	-7.27	<.0001*
Hg in Feed wt % TS	-0.073756	0.032089	-2.30	0.0319*

Residual by Predicted Plot

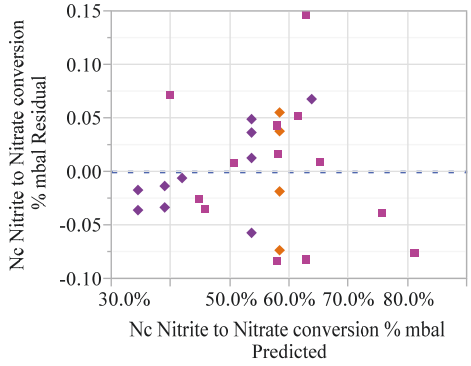
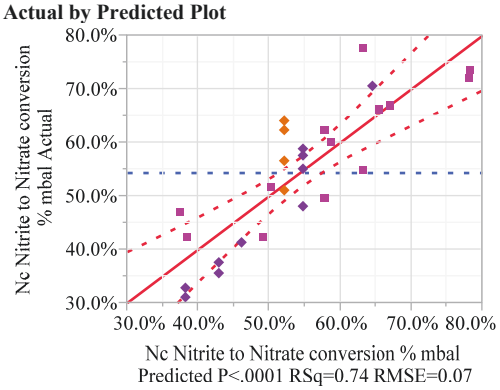


Exhibit N1b: Response Nc Nitrite to Nitrate conversion % mbal JMP Grouping=27 (SR+BH,NG)
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.737256
RSquare Adj	0.702985
Root Mean Square Error	0.070041
Mean of Response	0.543333
Observations (or Sum Wgts)	27

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	0.31660112	0.105534	21.5125
Error	23	0.11283088	0.004906	**Prob > F**
C. Total	26	0.42943200		<.0001*

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	15	0.09560163	0.006373	2.9594
Pure Error	8	0.01722925	0.002154	**Prob > F**
Total Error	23	0.11283088		0.0627
				Max RSq

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	1.3649127	0.381599	3.58	0.0016*
Acid Stoichiometry	0.8810754	0.13367	6.59	<.0001*
Percent Reducing Acid	-1.26796	0.462524	-2.74	0.0116*
Nitrite in Feed (before trims) mg/kg	-9.09e-5	0.000015	-6.10	<.0001*

Residual by Predicted Plot

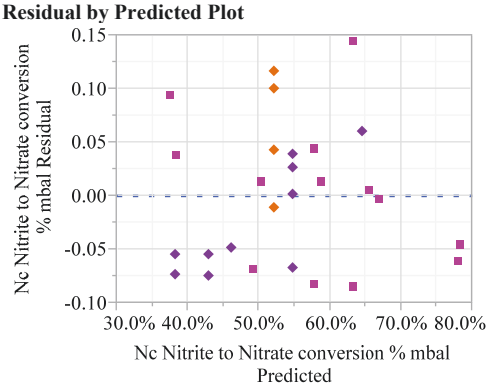
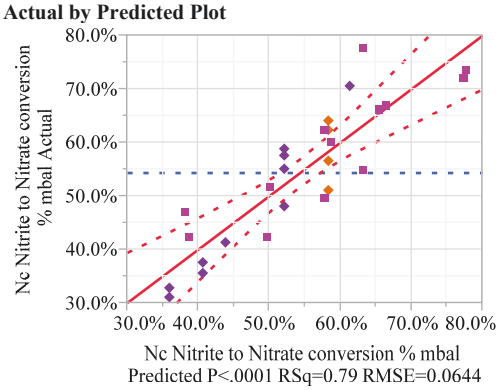


Exhibit N1c: Response Nc Nitrite to Nitrate conversion % mbal JMP Grouping=27 (SR+BH,NG)
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.787543
RSquare Adj	0.748915
Root Mean Square Error	0.064398
Mean of Response	0.543333
Observations (or Sum Wgts)	27

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	4	0.33819635	0.084549	20.3876
Error	22	0.09123565	0.004147	**Prob > F**
C. Total	26	0.42943200		<.0001*

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	14	0.07400640	0.005286	2.4545
Pure Error	8	0.01722925	0.002154	**Prob > F**
Total Error	22	0.09123565		0.1021
				Max RSq

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	1.8279261	0.4053	4.51	0.0002*
Acid Stoichiometry	0.8490422	0.1237	6.86	<.0001*
Percent Reducing Acid	-1.283672	0.425316	-3.02	0.0063*
Nitrite in Feed (before trims) mg/kg	-0.000114	0.000017	-6.71	<.0001*
Hg in Feed wt % TS	-0.07676	0.033638	-2.28	0.0325*

Residual by Predicted Plot

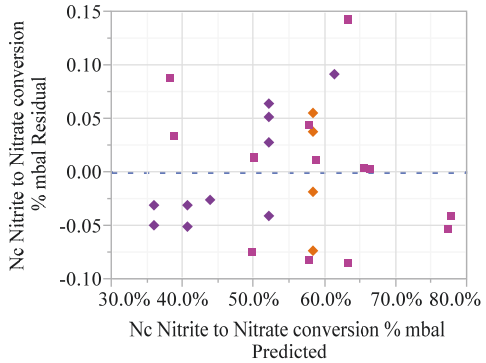
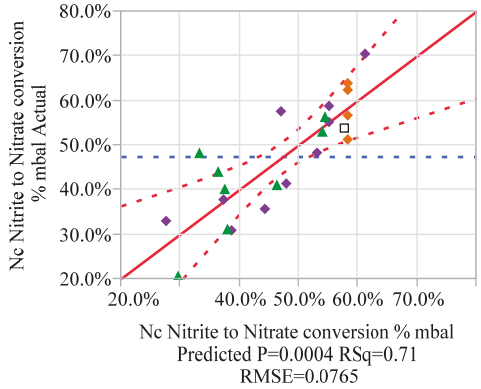


Exhibit N2a: Response Nc Nitrite to Nitrate conversion % mbal JMP Grouping=23 (SR+BH,43-50,57)
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.708769
RSquare Adj	0.623113
Root Mean Square Error	0.076476
Mean of Response	0.473739
Observations (or Sum Wgts)	23

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	5	0.24197267	0.048395	8.2746
Error	17	0.09942576	0.005849	**Prob > F**
C. Total	22	0.34139843		0.0004*

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	13	0.08858251	0.006814	2.5137
Pure Error	4	0.01084325	0.002711	**Prob > F**
Total Error	17	0.09942576		0.1933
				Max RSq

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	-1.46755	0.672009	-2.18	0.0433*
Acid Stoichiometry	1.5300382	0.294079	5.20	<.0001*
AS*PRA	-1.492062	0.48299	-3.09	0.0067*
HSV	0.1765425	0.089442	1.97	0.0649
Mn in Feed (before trims) wt % CS	0.1504669	0.09897	1.52	0.1468
Hg in Feed wt % TS	-0.05702	0.04241	-1.34	0.1965

Residual by Predicted Plot

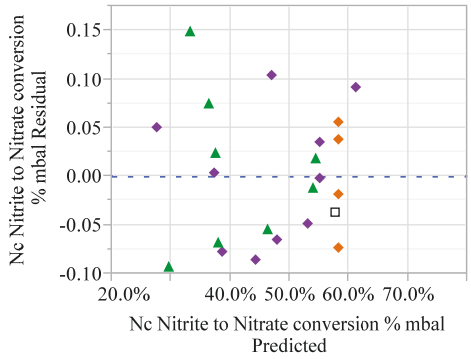
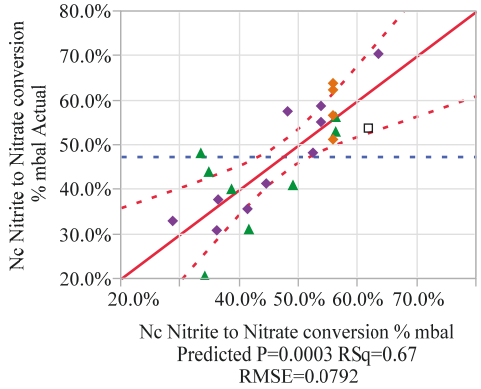


Exhibit N2b: Response Nc Nitrite to Nitrate conversion % mbal JMP Grouping=23 (SR+BH,43-50,57)
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.669172
RSquare Adj	0.595655
Root Mean Square Error	0.079213
Mean of Response	0.473739
Observations (or Sum Wgts)	23

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	4	0.22845436	0.057114	9.1022
Error	18	0.11294408	0.006275	**Prob > F**
C. Total	22	0.34139843		0.0003*

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	14	0.10210083	0.007293	2.6903
Pure Error	4	0.01084325	0.002711	**Prob > F**
Total Error	18	0.11294408		0.1751
				Max RSq

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	-0.540224	0.292147	-1.85	0.0809
Acid Stoichiometry	1.6459015	0.294198	5.59	<.0001*
AS*PRA	-1.330018	0.487942	-2.73	0.0139*
HSV	0.1221992	0.084923	1.44	0.1673
Hg in Feed wt % TS	-0.042313	0.04277	-0.99	0.3356

Residual by Predicted Plot

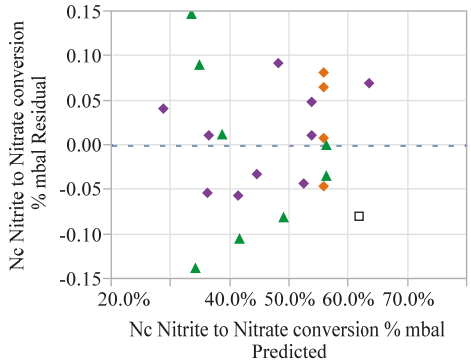
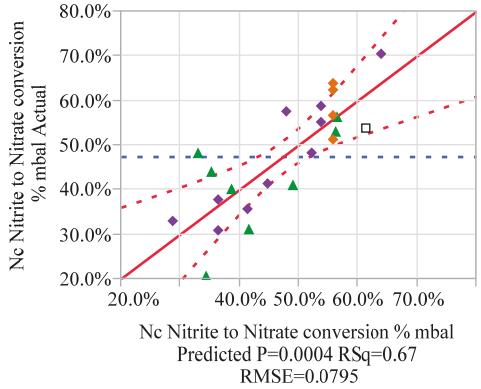


Exhibit N2c: Response Nc Nitrite to Nitrate conversion % mbal JMP Grouping=23 (SR+BH,43-50,57)
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.666616
RSquare Adj	0.592531
Root Mean Square Error	0.079518
Mean of Response	0.473739
Observations (or Sum Wgts)	23

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	4	0.22758182	0.056895	8.9980
Error	18	0.11381661	0.006323	**Prob > F**
C. Total	22	0.34139843		0.0004*

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	14	0.10297336	0.007355	2.7133
Pure Error	4	0.01084325	0.002711	**Prob > F**
Total Error	18	0.11381661		0.1729
				Max RSq

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.1785578	0.441976	0.40	0.6910
Acid Stoichiometry	0.9239048	0.221154	4.18	0.0006*
HSV	0.1243649	0.085195	1.46	0.1616
Hg in Feed wt % TS	-0.041695	0.042931	-0.97	0.3443
Percent Reducing Acid	-1.327954	0.493705	-2.69	0.0150*

Residual by Predicted Plot

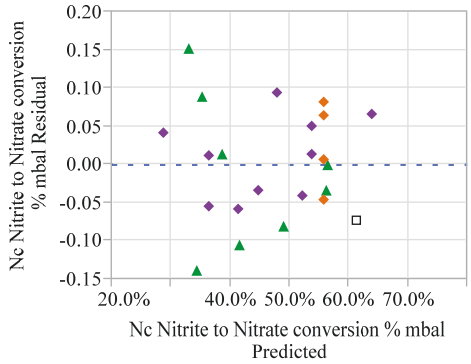
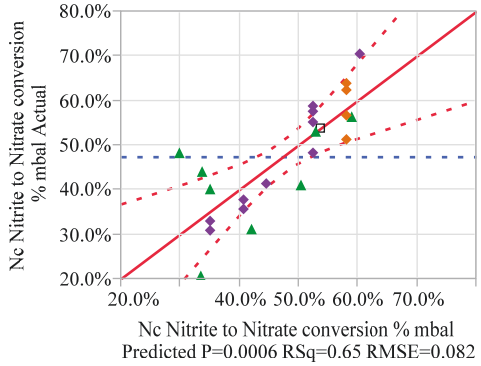


Exhibit N2d: Response Nc Nitrite to Nitrate conversion % mbal JMP Grouping=23 (SR+BH,43-50,57)
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.645567
RSquare Adj	0.566804
Root Mean Square Error	0.08199
Mean of Response	0.473739
Observations (or Sum Wgts)	23

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	4	0.22039544	0.055099	8.1963
Error	18	0.12100299	0.006722	**Prob > F**
C. Total	22	0.34139843		0.0006*

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	10	0.10377374	0.010377	4.8185
Pure Error	8	0.01722925	0.002154	**Prob > F**
Total Error	18	0.12100299		0.0178*
				Max RSq

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.2124924	0.488881	0.43	0.6690
Acid Stoichiometry	0.8123102	0.217057	3.74	0.0015*
Hg in Feed wt % TS	-0.075809	0.040418	-1.88	0.0770
Percent Reducing Acid	-1.532496	0.532339	-2.88	0.0100*
Nitrite in Feed (before trims) mg/kg	3.0846e-5	3.189e-5	0.97	0.3463

Residual by Predicted Plot

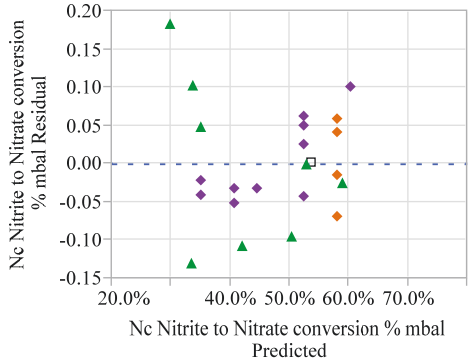
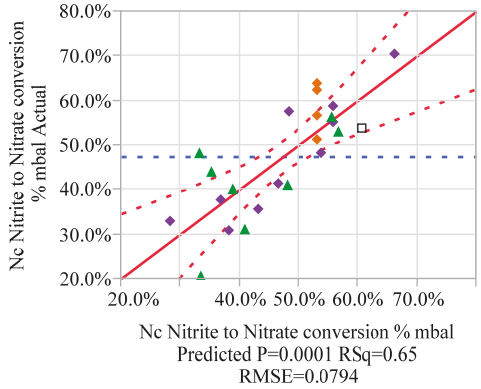


Exhibit N2e: Response Nc Nitrite to Nitrate conversion % mbal JMP Grouping=23 (SR+BH,43-50,57)
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.649146
RSquare Adj	0.593748
Root Mean Square Error	0.079399
Mean of Response	0.473739
Observations (or Sum Wgts)	23

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	0.22161752	0.073873	11.7179
Error	19	0.11978092	0.006304	**Prob > F**
C. Total	22	0.34139843		0.0001*

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	15	0.10893767	0.007263	2.6791
Pure Error	4	0.01084325	0.002711	**Prob > F**
Total Error	19	0.11978092		0.1759
				Max RSq

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.0482862	0.420501	0.11	0.9098
Acid Stoichiometry	0.9427072	0.219976	4.29	0.0004*
Percent Reducing Acid	-1.320454	0.492906	-2.68	0.0148*
HSV	0.1605249	0.076515	2.10	0.0495*

Residual by Predicted Plot

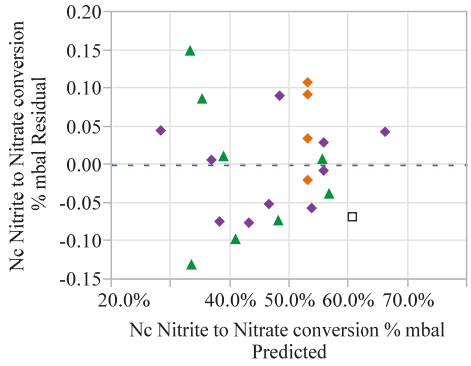
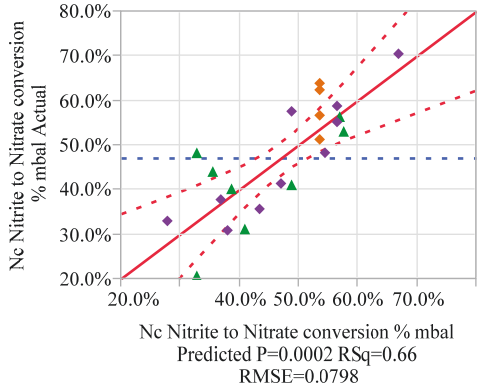


Exhibit N2f: Response Nc Nitrite to Nitrate conversion % mbal JMP Grouping=22 (SR+BH,43-50)
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.660176
RSquare Adj	0.603538
Root Mean Square Error	0.079757
Mean of Response	0.470773
Observations (or Sum Wgts)	22

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	0.22244343	0.074148	11.6562
Error	18	0.11450243	0.006361	**Prob > F**
C. Total	21	0.33694586		0.0002*

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	14	0.10365918	0.007404	2.7314
Pure Error	4	0.01084325	0.002711	**Prob > F**
Total Error	18	0.11450243		0.1713
				Max RSq

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.0971886	0.425795	0.23	0.8220
Acid Stoichiometry	0.9628171	0.222068	4.34	0.0004*
Percent Reducing Acid	-1.452846	0.516019	-2.82	0.0115*
HSV	0.1658022	0.077078	2.15	0.0453*

Residual by Predicted Plot

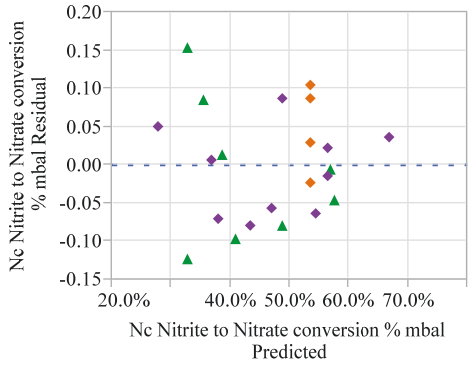
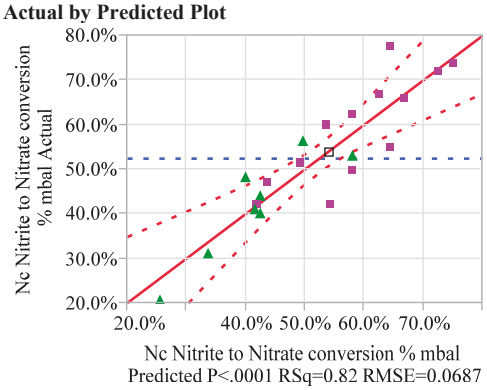


Exhibit N3a: Response Nc Nitrite to Nitrate conversion % mbal JMP Grouping=22 (NG,43-50,57)
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.816702
RSquare Adj	0.759422
Root Mean Square Error	0.0687
Mean of Response	0.524273
Observations (or Sum Wgts)	22

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	5	0.33646036	0.067292	14.2579
Error	16	0.07551401	0.004720	**Prob > F**
C. Total	21	0.41197436		<.0001*

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	-1.270591	0.468024	-2.71	0.0153*
Acid Stoichiometry	0.6305583	0.141482	4.46	0.0004*
Percent Reducing Acid	-1.453491	0.363964	-3.99	0.0010*
SASV	18.112092	5.676497	3.19	0.0057*
HSV	-6.312573	2.062654	-3.06	0.0075*
Fe in Feed (before trims) wt % CS	-0.003139	0.019832	-0.16	0.8762

Residual by Predicted Plot

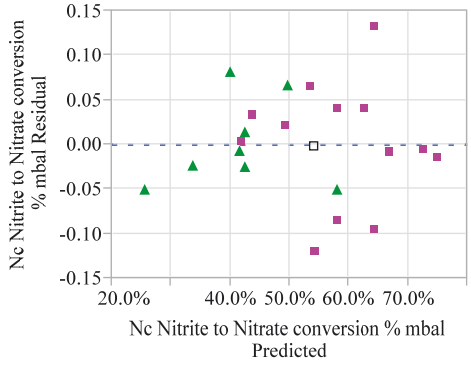
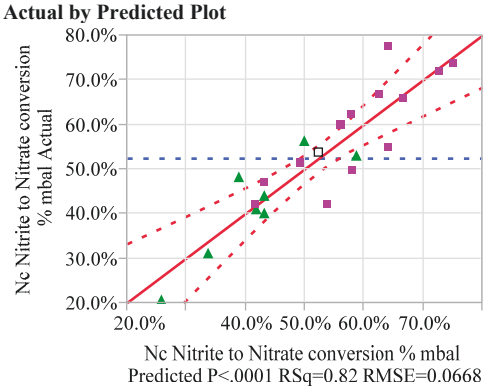


Exhibit N3b: Response Nc Nitrite to Nitrate conversion % mbal JMP Grouping=22 (NG,43-50,57)
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.815625
RSquare Adj	0.772243
Root Mean Square Error	0.066844
Mean of Response	0.524273
Observations (or Sum Wgts)	22

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	4	0.33601677	0.084004	18.8009
Error	17	0.07595759	0.004468	**Prob > F**
C. Total	21	0.41197436		<.0001*

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	-0.651016	0.360644	-1.81	0.0888
Acid Stoichiometry	0.6409423	0.135541	4.73	0.0002*
Percent Reducing Acid	-1.451289	0.34378	-4.22	0.0006*
HSV	0.4756765	0.146539	3.25	0.0048*
Hg in Feed wt % TS	0.4559099	0.086459	5.27	<.0001*

Residual by Predicted Plot

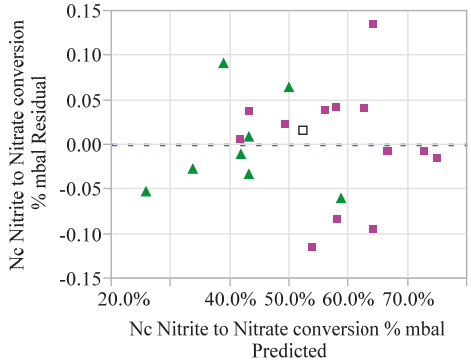
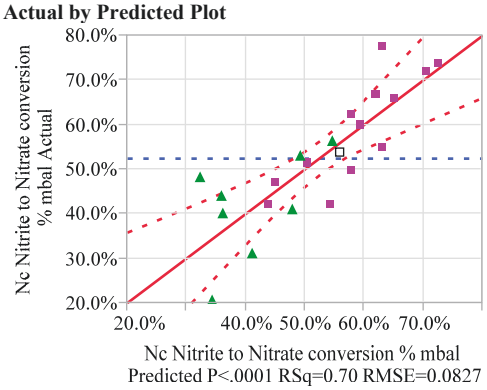


Exhibit N3c: Response Nc Nitrite to Nitrate conversion % mbal JMP Grouping=22 (NG,43-50,57)
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.701345
RSquare Adj	0.651569
Root Mean Square Error	0.082677
Mean of Response	0.524273
Observations (or Sum Wgts)	22

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	0.28893621	0.096312	14.0901
Error	18	0.12303815	0.006835	**Prob > F**
C. Total	21	0.41197436		<.0001*

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.2525248	0.283635	0.89	0.3850
Acid Stoichiometry	0.5542686	0.16436	3.37	0.0034*
Percent Reducing Acid	-1.220217	0.415994	-2.93	0.0089*
Hg in Feed wt % TS	0.1952079	0.0396	4.93	0.0001*

Residual by Predicted Plot

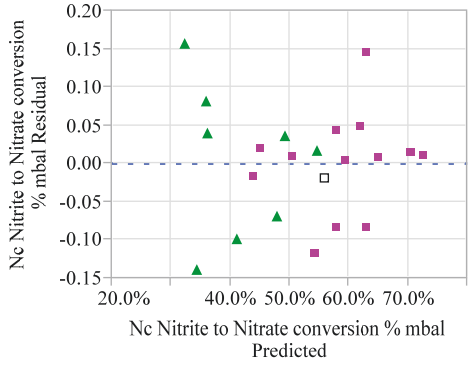
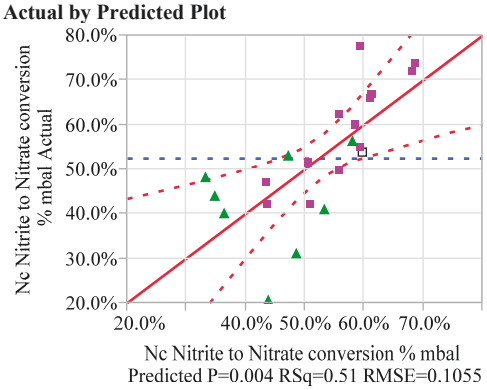


Exhibit N3d: Response Nc Nitrite to Nitrate conversion % mbal JMP Grouping=22 (NG,43-50,57)
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.514056
RSquare Adj	0.433065
Root Mean Square Error	0.105461
Mean of Response	0.524273
Observations (or Sum Wgts)	22

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	0.21177769	0.070593	6.3471
Error	18	0.20019667	0.011122	**Prob > F**
C. Total	21	0.41197436		0.0040*

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.6745341	0.407997	1.65	0.1156
Acid Stoichiometry	0.5392186	0.211669	2.55	0.0202*
Percent Reducing Acid	-0.854657	0.512171	-1.67	0.1125
HSV	-0.242107	0.085614	-2.83	0.0111*

Residual by Predicted Plot

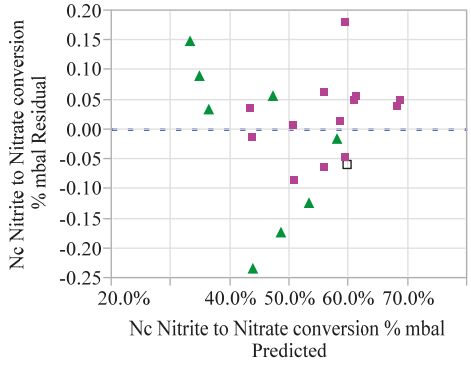
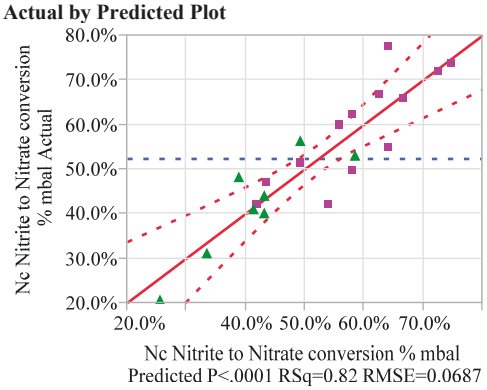


Exhibit N3e: Response Nc Nitrite to Nitrate conversion % mbal JMP Grouping=21 (NG,43-50)
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.816343
RSquare Adj	0.770429
Root Mean Square Error	0.068748
Mean of Response	0.523571
Observations (or Sum Wgts)	21

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	4	0.33612687	0.084032	17.7797
Error	16	0.07562027	0.004726	**Prob > F**
C. Total	20	0.41174714		<.0001*

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	-0.674567	0.381248	-1.77	0.0959
Acid Stoichiometry	0.6339578	0.141832	4.47	0.0004*
Percent Reducing Acid	-1.431031	0.361612	-3.96	0.0011*
HSV	0.4830467	0.153217	3.15	0.0062*
Hg in Feed wt % TS	0.4616286	0.091462	5.05	0.0001*

Residual by Predicted Plot

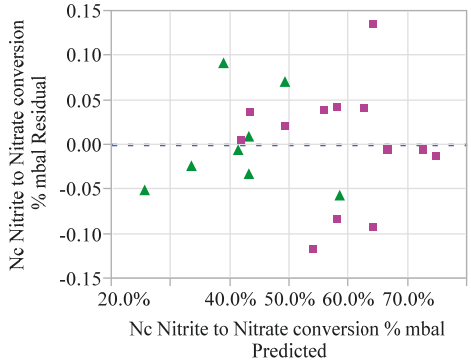
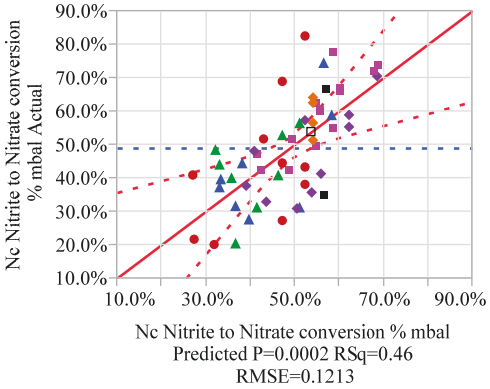


Exhibit N4a: Response Nc Nitrite to Nitrate conversion % mbal JMP Grouping=ALL (56)
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.461266
RSquare Adj	0.369567
Root Mean Square Error	0.121274
Mean of Response	0.489089
Observations (or Sum Wgts)	56

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	8	0.5918485	0.073981	5.0302
Error	47	0.6912461	0.014707	**Prob > F**
C. Total	55	1.2830946		0.0002*

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	40	0.49478480	0.012370	0.4407
Pure Error	7	0.19646125	0.028066	**Prob > F**
Total Error	47	0.69124605		0.9518
				Max RSq

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	-0.847239	0.328282	-2.58	0.0130*
Acid Stoichiometry	1.0917038	0.269036	4.06	0.0002*
AS*PRA	-0.881895	0.483946	-1.82	0.0748
SASV	0.7293465	0.308873	2.36	0.0224*
HSV	-0.312613	0.185812	-1.68	0.0991
Nitrite in Feed (before trims) mg/kg	1.929e-5	1.616e-5	1.19	0.2387
Fe in Feed (before trims) wt % CS	0.0084761	0.003717	2.28	0.0272*
Mn in Feed (before trims) wt % CS	0.026485	0.017346	1.53	0.1335
Hg in Feed wt % TS	0.0540964	0.033393	1.62	0.1119

Residual by Predicted Plot

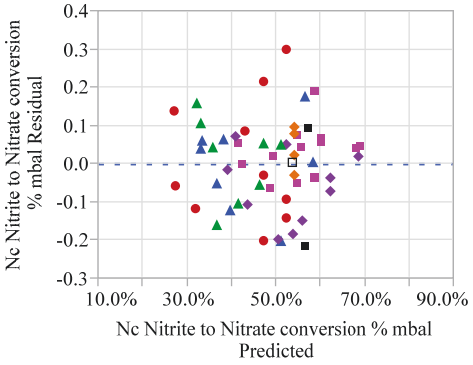
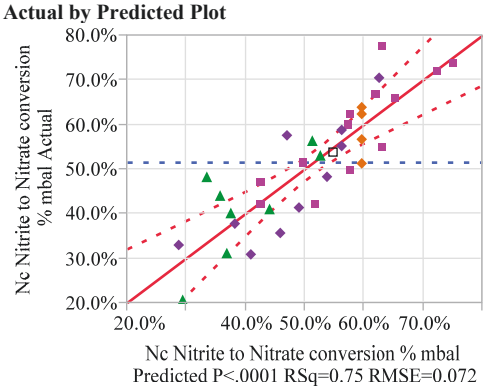


Exhibit N4b: Response Nc Nitrite to Nitrate conversion % mbal JMP Grouping=36 (SR+BH,NG,43-50,57)
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.74993
RSquare Adj	0.708251
Root Mean Square Error	0.07205
Mean of Response	0.515278
Observations (or Sum Wgts)	36

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	5	0.46702717	0.093405	17.9933
Error	30	0.15573405	0.005191	**Prob > F**
C. Total	35	0.62276122		<.0001*

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	26	0.14489080	0.005573	2.0557
Pure Error	4	0.01084325	0.002711	**Prob > F**
Total Error	30	0.15573405		0.2545
				Max RSq

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	-1.96528	0.322242	-6.10	<.0001*
Acid Stoichiometry	1.408325	0.206266	6.83	<.0001*
AS*PRA	-1.30379	0.335493	-3.89	0.0005*
HSV	0.1980516	0.080145	2.47	0.0194*
Mn in Feed (before trims) wt % CS	0.2178971	0.033493	6.51	<.0001*
Hg in Feed wt % TS	-0.055834	0.039387	-1.42	0.1666

Residual by Predicted Plot

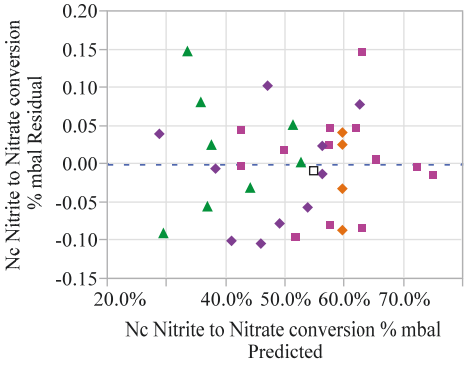
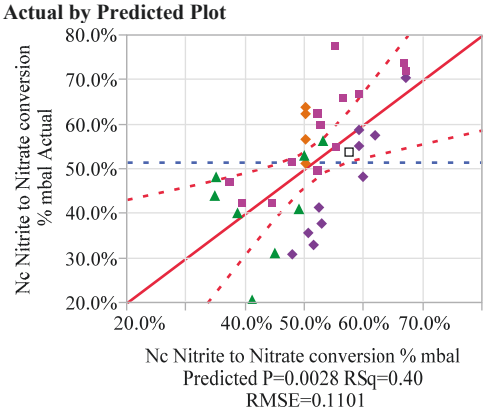


Exhibit N4c: Response Nc Nitrite to Nitrate conversion % mbal JMP Grouping=36 (SR+BH,NG,43-50,57)
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.397124
RSquare Adj	0.319333
Root Mean Square Error	0.110051
Mean of Response	0.515278
Observations (or Sum Wgts)	36

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	4	0.24731332	0.061828	5.1050
Error	31	0.37544790	0.012111	**Prob > F**
C. Total	35	0.62276122		0.0028*

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	27	0.36460465	0.013504	4.9815
Pure Error	4	0.01084325	0.002711	**Prob > F**
Total Error	31	0.37544790		0.0642
				Max RSq

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	-0.246684	0.281879	-0.88	0.3882
Acid Stoichiometry	1.0653184	0.304592	3.50	0.0014*
AS*PRA	-0.71853	0.493678	-1.46	0.1556
HSV	-0.058517	0.106571	-0.55	0.5869
Hg in Feed wt % TS	0.0648539	0.05307	1.22	0.2309

Residual by Predicted Plot

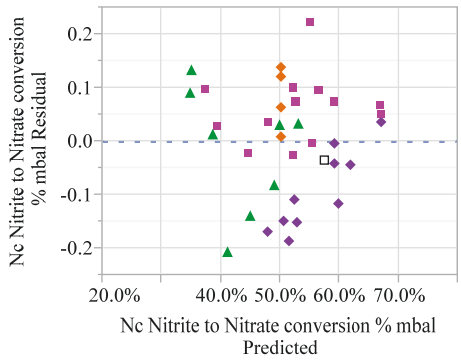
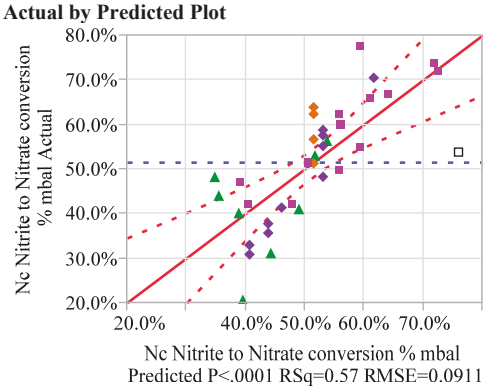


Exhibit N4d: Response Nc Nitrite to Nitrate conversion % mbal JMP Grouping=36 (SR+BH,NG,43-50,57)
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.573838
RSquare Adj	0.533885
Root Mean Square Error	0.09107
Mean of Response	0.515278
Observations (or Sum Wgts)	36

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	0.35736402	0.119121	14.3629
Error	32	0.26539720	0.008294	**Prob > F**
C. Total	35	0.62276122		<.0001*

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	24	0.24816795	0.010340	4.8013
Pure Error	8	0.01722925	0.002154	**Prob > F**
Total Error	32	0.26539720		0.0136*
				Max RSq

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	1.0453999	0.36331	2.88	0.0071*
Acid Stoichiometry	0.7285175	0.151716	4.80	<.0001*
Percent Reducing Acid	-0.855349	0.409987	-2.09	0.0450*
Nitrite in Feed (before trims) mg/kg	-7.017e-5	1.527e-5	-4.59	<.0001*

Residual by Predicted Plot

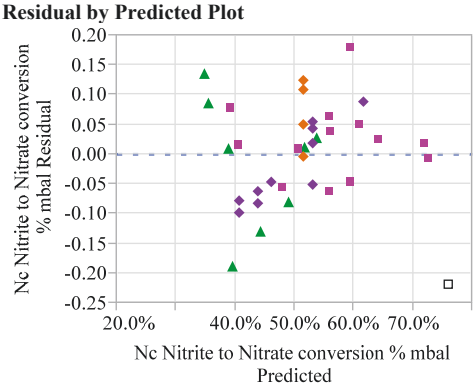
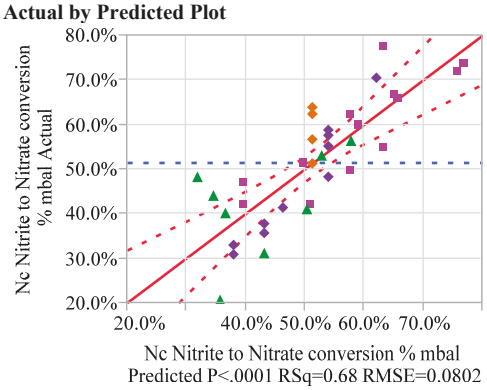


Exhibit N4e: Response Nc Nitrite to Nitrate conversion % mbal JMP Grouping=35 (SR+BH,NG,43-50)
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.679844
RSquare Adj	0.648861
Root Mean Square Error	0.08016
Mean of Response	0.5146
Observations (or Sum Wgts)	35

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	0.42298701	0.140996	21.9426
Error	31	0.19919539	0.006426	**Prob > F**
C. Total	34	0.62218240		<.0001*

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	23	0.18196614	0.007912	3.6736
Pure Error	8	0.01722925	0.002154	**Prob > F**
Total Error	31	0.19919539		0.0313*
				Max RSq

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	1.4907745	0.348594	4.28	0.0002*
Acid Stoichiometry	0.7862982	0.13475	5.84	<.0001*
Percent Reducing Acid	-1.326445	0.389578	-3.40	0.0018*
Nitrite in Feed (before trims) mg/kg	-9.075e-5	1.489e-5	-6.09	<.0001*

Residual by Predicted Plot

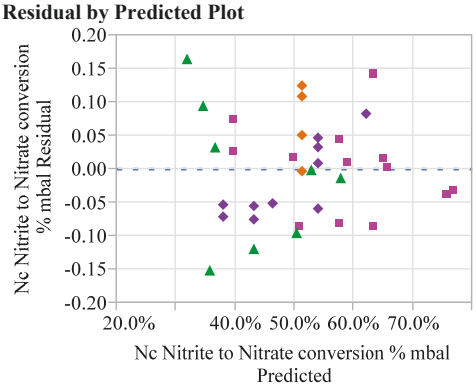
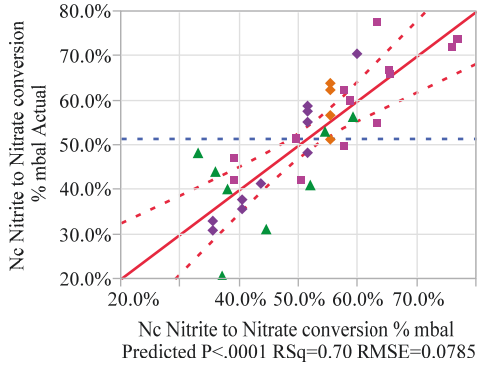


Exhibit N4f: Response Nc Nitrite to Nitrate conversion % mbal JMP Grouping=35 (SR+BH,NG,43-50)
Whole Model
Actual by Predicted Plot



Summary of Fit

RSquare	0.702814
RSquare Adj	0.66319
Root Mean Square Error	0.078508
Mean of Response	0.5146
Observations (or Sum Wgts)	35

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	4	0.43727867	0.109320	17.7367
Error	30	0.18490373	0.006163	**Prob > F**
C. Total	34	0.62218240		<.0001*

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	22	0.16767448	0.007622	3.5389
Pure Error	8	0.01722925	0.002154	**Prob > F**
Total Error	30	0.18490373		0.0352*
				Max RSq

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	1.8237901	0.405446	4.50	<.0001*
Acid Stoichiometry	0.8015784	0.132353	6.06	<.0001*
Percent Reducing Acid	-1.327038	0.381547	-3.48	0.0016*
Nitrite in Feed (before trims) mg/kg	-0.000111	1.989e-5	-5.60	<.0001*
Hg in Feed wt % TS	-0.056038	0.036801	-1.52	0.1383

Residual by Predicted Plot

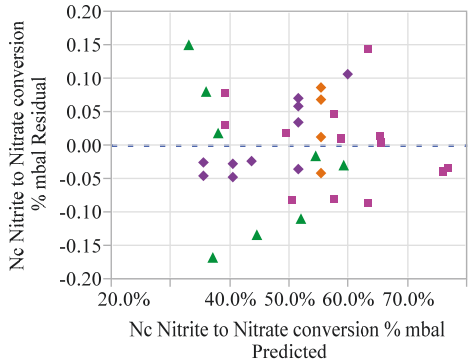
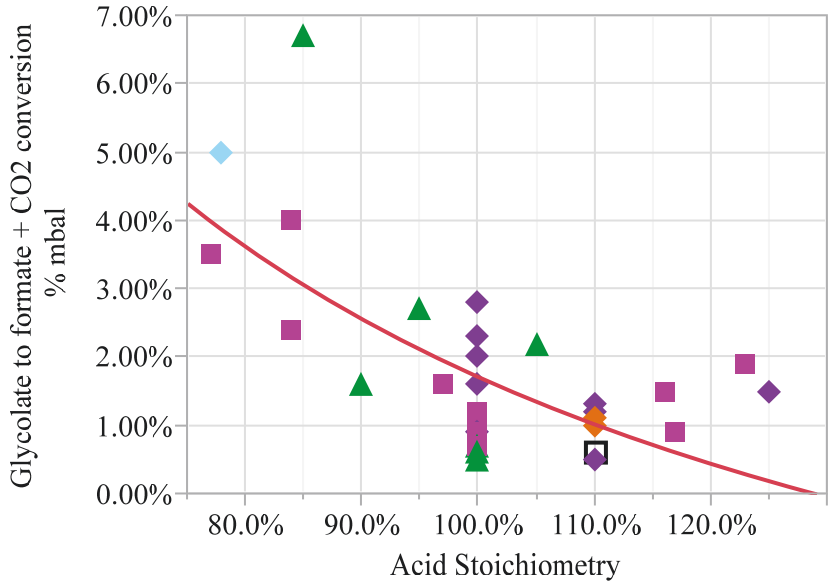


Exhibit GtoF. Fit of glycolate to formate + CO_2 (Refer to Equation 20)
Bivariate Fit of Glycolate to formate + CO2 conversion % mbal By Acid Stoichiometry JMP Grouping=37S *(SR+BH,NG,43-50,57,SC-18)



Transformed Fit to Reciprocal

Glycolate to formate + CO2 conversion % mbal = -0.059033 + 0.076181*Recip(Acid Stoichiometry)

Summary of Fit

RSquare	0.4398
RSquare Adj	0.423794
Root Mean Square Error	0.01006
Mean of Response	0.01673
Observations (or Sum Wgts)	37

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	12	0.00266071	0.000222	5.7846
Pure Error	23	0.00088160	0.000038	**Prob > F**
Total Error	35	0.00354231		0.0002*
				Max RSq

Analysis of Variance

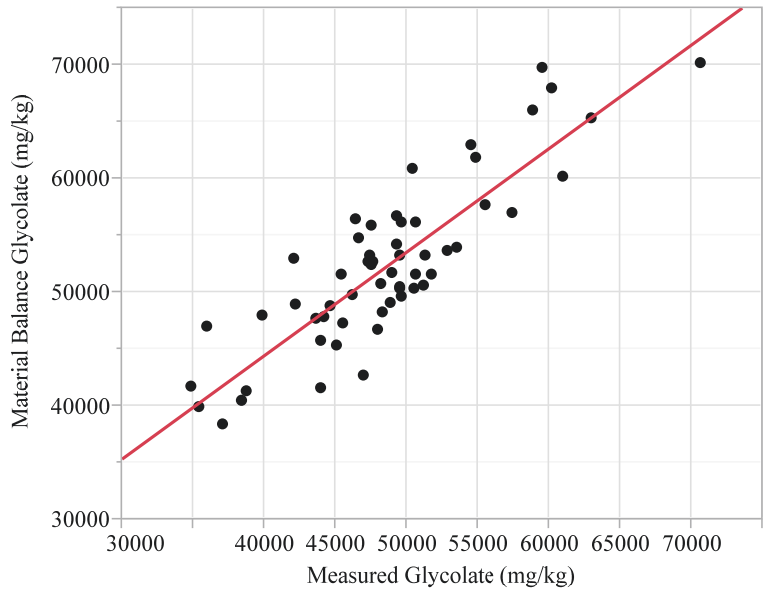
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	1	0.00278098	0.002781	27.4777
Error	35	0.00354231	0.000101	**Prob > F**
C. Total	36	0.00632330		<.0001*

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	-0.059033	0.014548	-4.06	0.0003*
Recip(Acid Stoichiometry)	0.076181	0.014533	5.24	<.0001*

Exhibit Reference to Figure 1: Bivariate Fit of Material Balance Glycolate (mg/kg) By Measured Glycolate (mg/kg)

(Refer to Figure 1)



Linear Fit

Material Balance Glycolate (mg/kg) = 7961.989 + 0.9113491*Measured Glycolate (mg/kg)

Summary of Fit

RSquare	0.748119
RSquare Adj	0.743539
Root Mean Square Error	3680.826
Mean of Response	52295
Observations (or Sum Wgts)	57

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	53	739768353	13957893	5.1716
Pure Error	2	5397875	2698937.3	**Prob > F**
Total Error	55	745166227		0.1752
				Max RSq

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	1	2213240287	2.2132e+9	163.3571
Error	55	745166227	13548477	**Prob > F**
C. Total	56	2958406514		<.0001*

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	7961.989	3502.727	2.27	0.0269*
Measured Glycolate (mg/kg)	0.9113491	0.071304	12.78	<.0001*

Appendix C Tables of Regression Coefficients for Preferred Models

Regression coefficients are shown in **bold**. The standard error of each regression coefficient is shown below the value in ***bold italics*** and the P-value in standard text.

Value
± *Std Error*
P-value

Glycolate Destruction (G_D) Model G1 (R^2=0.69):

Intercept	AS (%)*	Nitrate (mg/kg)	Hg (wt % TS)
0.2997349 ***0.0477770*** <0.0001	**-0.319911** ***0.039680*** <0.0001	**1.9179E-5** ***0.3943E-5*** <0.0001	**0.0232576** ***0.0086740*** 0.0115

* e.g., 110% would be 1.10

Glycolate to Oxalate Conversion (GtoOx) Models X6 (R^2=0.77) and X7 (R^2=0.68):

Model	Intercept	AS (%)	Nitrite (mg/kg in Feed)	Ru-Nitrosyl Present (Y/N)
X6	**0.4459707** ***0. 037856*** <0.0001	**-0.187849** ***0.024877*** <0.0001	**-1.8754E-5** ***2.485E-6*** <0.0001	**NA**
X7	**0.2182302** ***0.030481*** <0.0001	**-0.170654** ***0.029893*** <0.0001	**NA**	**-0.01870** (Yes: add 0.0187; No: subtract 0.0187) ***0.00341*** <0.0001

Glycolate to Formate Conversion (GtoF) *with* Noble Metals and Hg Present (R^2=0.44):

Intercept	AS^{-1}
-0.059033 ***0.014548*** 0.0003	**0.076181** ***0.014533*** <0.0001

Nitrite to Nitrate Conversion (N_C) Models N4e (R^2=0.68) and N4f (R^2=0.70):

Model	Intercept	AS (%)	PRA (%)	Nitrite (mg/kg)	Hg (wt % TS)
N4e	**1.4908845** ***0.3485940*** 0.0002	**0.7862982** ***0.1347500*** <0.0001	**-1.326445** ***0.389578*** 0.0018	**-9.075E-5** ***1.489E-5*** <0.0001	NA
N4f	**1.8237901** ***0.4054460*** <0.0001	**0.8015784** ***0.1323530*** <0.0001	**-1.327038** ***0.381547*** 0.0016	**1.1100E-4** ***0.1989E-4*** <0.0001	**-0.056038** ***0.036801*** 0.1383

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