



July 25, 2017

Great Salt Lake Bioassays

Update and Summary of preliminary analytical trials using copper

Purpose: In the previous acute range-finding test conducted by North Carolina State University (NCSU), the dose-response observed was consistent with nominal copper concentrations. However, the analytical results did not correlate well with nominal concentrations. TRE conducted a similar experiment but no brine shrimp were used and two concentrations were spiked.

Methods: Reconstituted Great Salt Lake water (rGSL) was spiked in triplicate to two nominal concentrations, 375 µg/L and 1,750 µg/L. In one of the replicates, algae were added to the rGSL. Copper concentrations were measured at time-zero (T1) and after 24 hours (T2) for both dissolved (<0.45 µm) and total recoverable. At the conclusion of the test, the test vessels were rinsed with acid and the resulting rinsate analyzed for total recoverable copper. The stock solution was not analyzed.

Results: 58% to 74% of nominal copper concentration was measured in total recoverable copper analyses (Figures 1a and 1b). The dissolved portion of the total recoverable copper ranged from 8 to 84%. The amount of copper recovered increased in the 375 µg/L treatment when compared to the 1,750 µg/L treatment. Recoveries for both total recoverable and dissolved copper were higher in algae present treatments than in the no algae treatments.

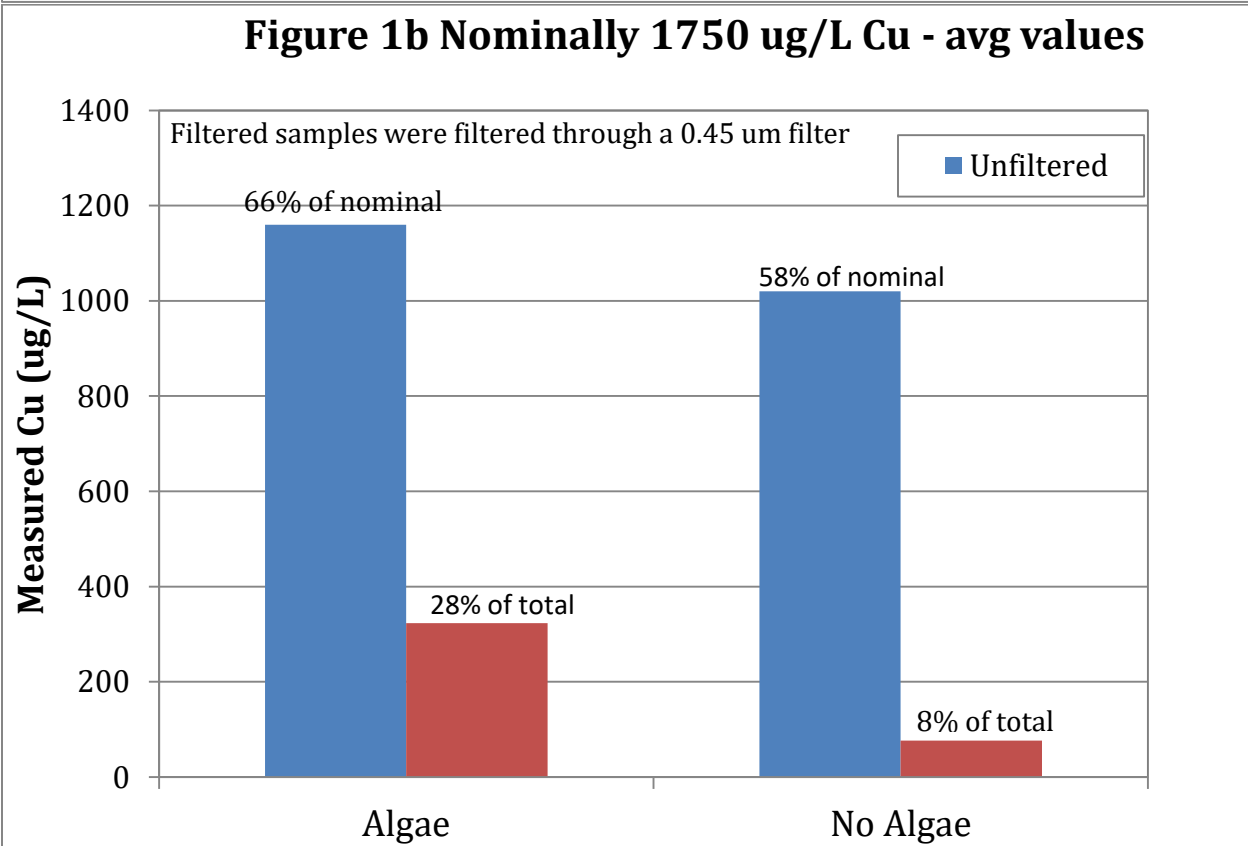
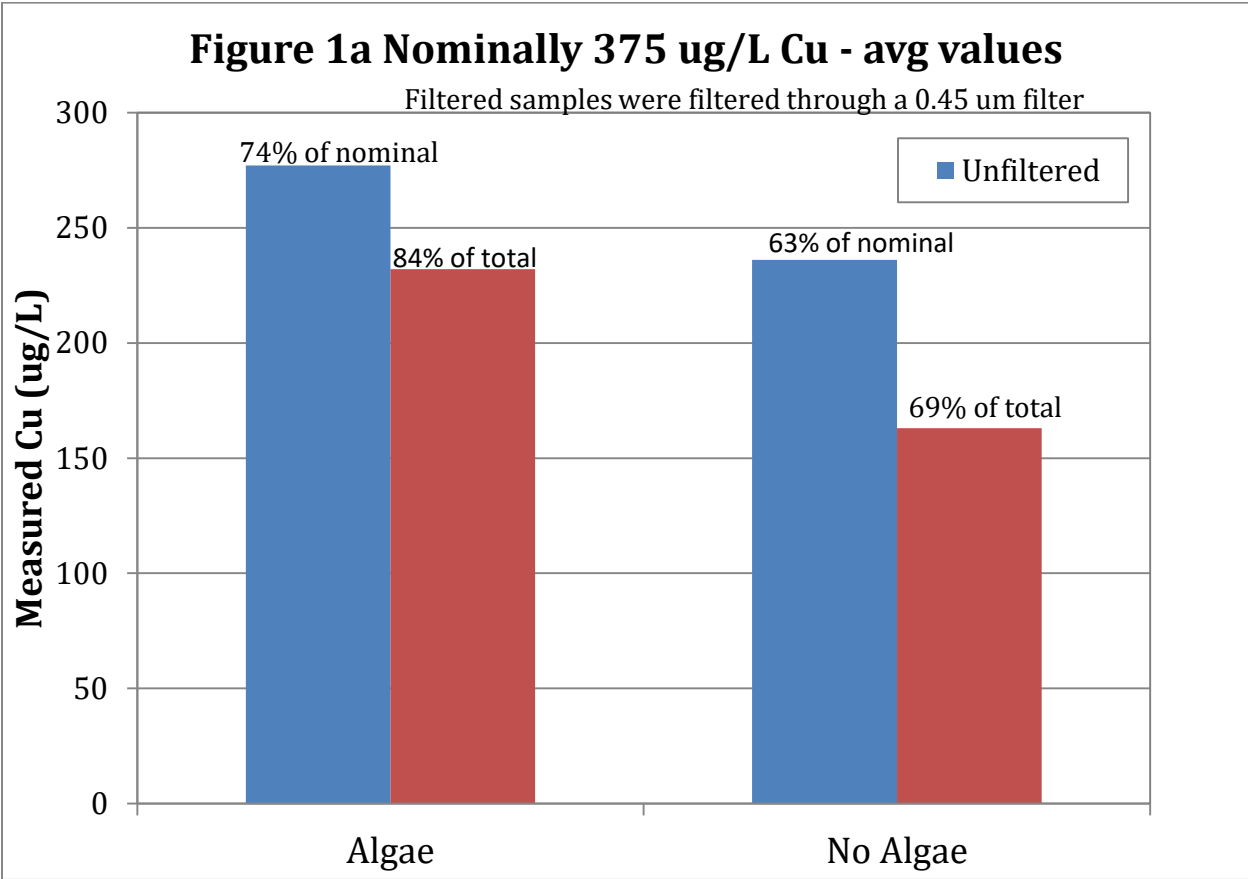
Figure 2a plots observed versus nominal dissolved copper with algae present for both the NCSU and TRE experiments. NCSU used a 0.22 µm filter whereas TRE used a 0.45 µm filter. Both NCSU and TRE observed lower recoveries at the higher spiked concentration. TRE's higher observed recoveries are likely attributable to the larger filter size.

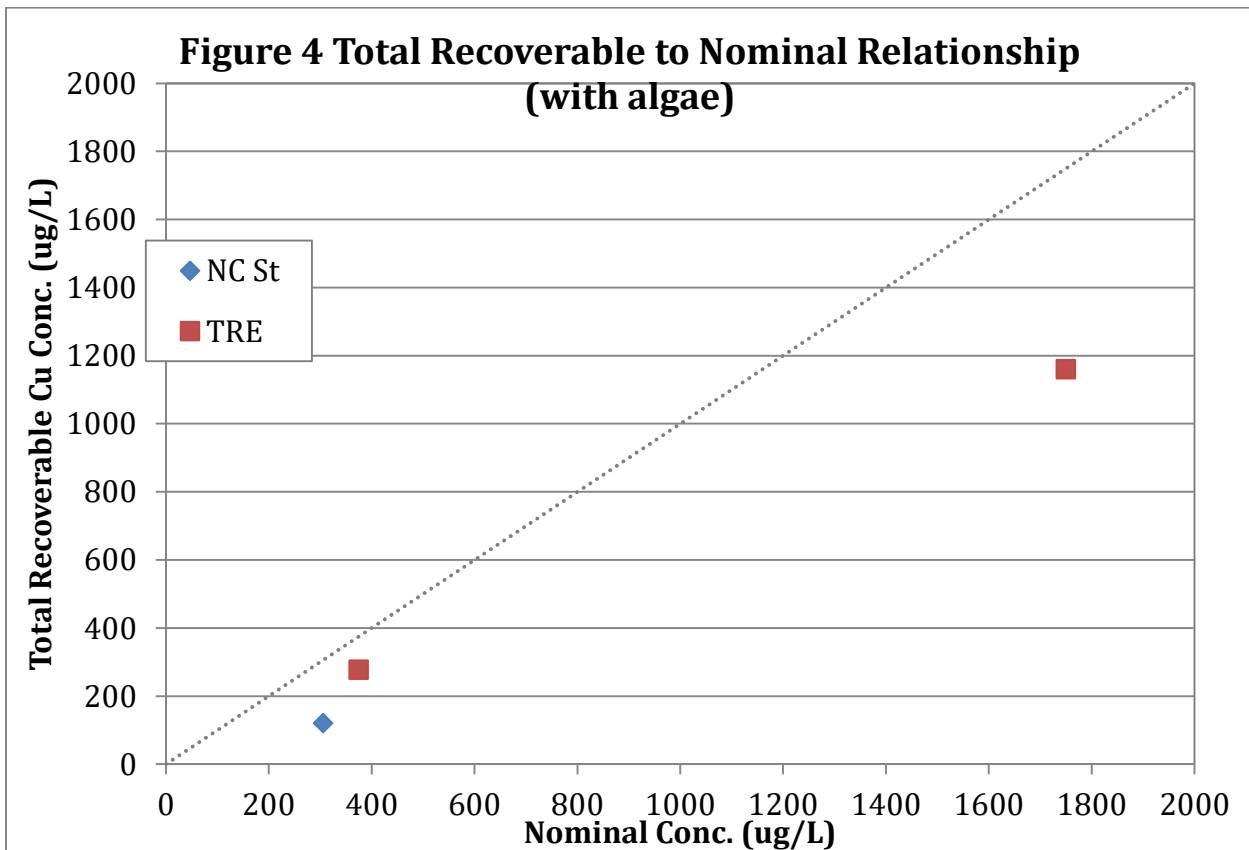
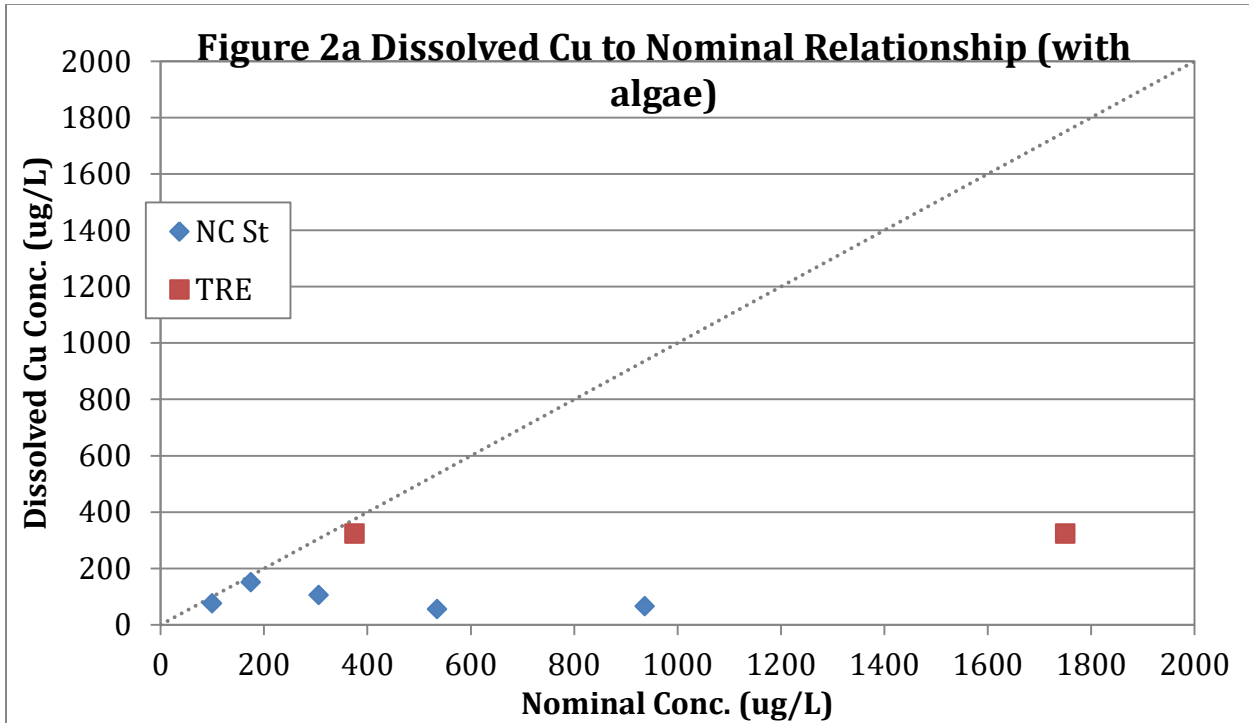
Figure 2b plots total recoverable copper versus nominal with algae present. Only one total recoverable metals sample was analyzed by NCSU. Compared to nominal concentrations, total recoverable copper had similar total recoveries at both 375 µg/L and 1,750 µg/L. Dissolved copper recoveries decrease markedly at the higher concentrations.

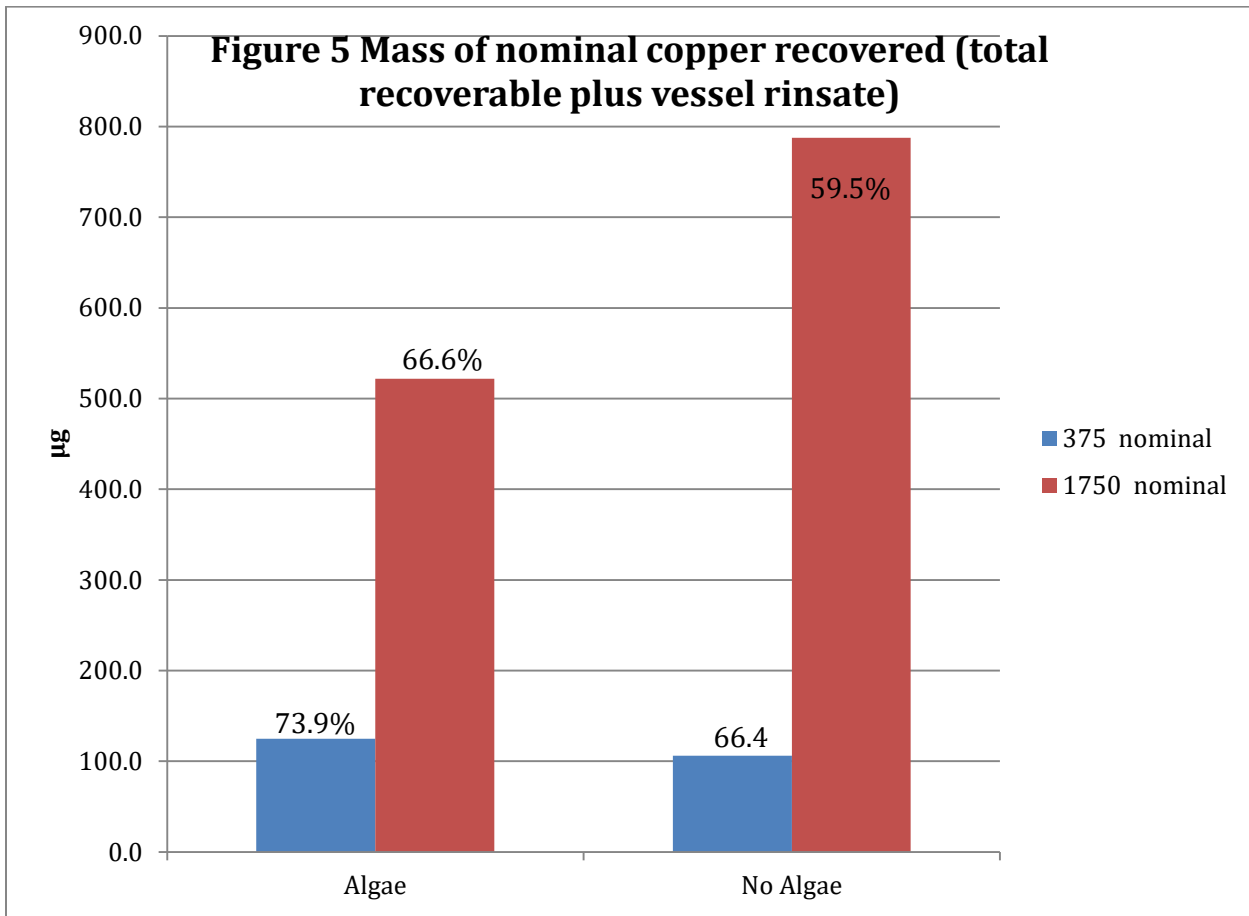
Figure 5¹ shows a total recoverable copper mass balance including copper residues extracted from the rinsate and samples taken from the test vessel. Total recoverable copper mass ranged from 59.5 to 73.9% of nominal values. Copper measured in the vessel rinsate was 5% or less of the total mass of copper recovered.

Discussion: Recoveries of the dissolved spiked copper were improved with using a 0.45 µm filter and was 84% of total recoverable copper and 62% of nominal concentrations for 375 µg/L with algae. Based on previous experiments, 400 µg/L is anticipated to be the approximate LC₅₀. This suggests that an acute test can be successfully completed.

¹ Figures were numbered the same as in the companion workbook [GSL_Cu_anal_trialMay2017.xlsx](#)







Summary:

TRE has completed a definitive 96-hr test for copper (Cu). The Utah Division of Water Quality (DWQ) proposes to direct TRE to complete the Cu analytical analyses and is seeking EPA's concurrence. The samples are scheduled to be analyzed on February 8, 2018.

Cu Results

Table 1 shows the nominal dilution series for the Cu definitive test. Figure 1 shows percent survival graphed against nominal Cu concentrations for this test. The resulting Cu 96-hr LC₅₀ is 966 µg/L nominal.

Nominal Cu Concentration (µg/L)	% Survival 96 hrs
Control	100
288	100
412	99
588	98
840	92
1,200	0

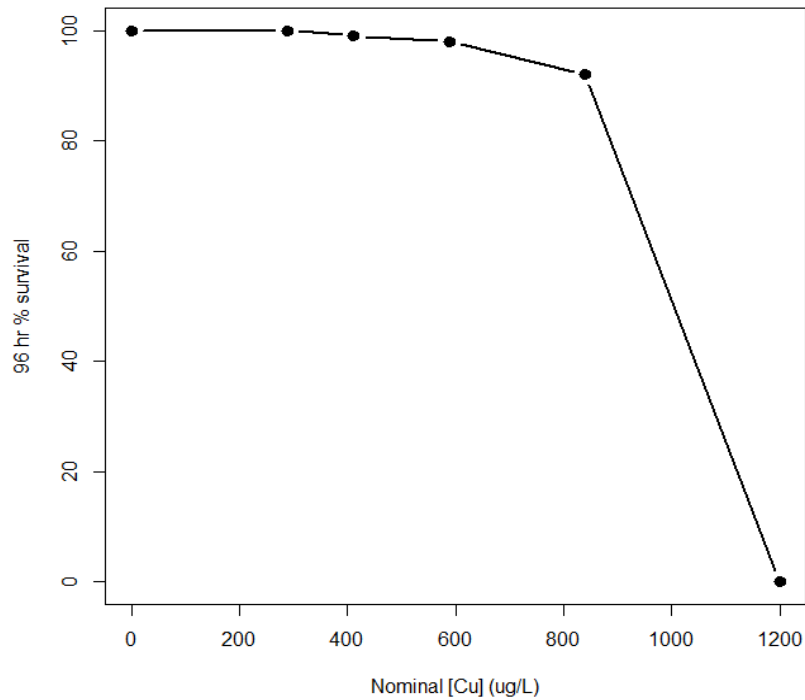


Figure 1 Brine shrimp survival and nominal copper concentrations

Unfortunately, none of the Cu dilutions resulted in survival rates of more than 10% and less than 90%. However, the results of the previous range-finding tests shown on Figure 2 are consistent with the responses observed for the definitive test. The consistency in dose-response supports that the LC₅₀ can be interpolated for the definitive test. The causes of the anomalous September 13, 2017 result showing complete mortality at about 300 µg/L nominal Cu are unknown.

Figures 3 and 4 show all available measured dissolved Cu concentrations (<0.45 µM) and total recoverable Cu concentrations versus nominal concentrations, respectively. The orange circles represent results of samples analyzed from the definitive test. Only the nominal 840 and 1,200 µg/L treatments were analyzed to confirm the nominal concentrations between the two treatments where survival transitioned from 92% to 0%. The remaining samples are set to be analyzed for both total and dissolved Cu.

Figures 3 and 4 indicate good precision between the different range-finding tests. As shown on Figure 3, the dissolved Cu concentrations appear to plateau at around 300 µg/L, above which the measured dissolved Cu concentrations do not increase with increasing nominal concentrations. As shown on Figure 4, the total recoverable Cu concentrations did increase with increasing nominal concentrations. Based on the dose-response observed with nominal concentrations in Figure 1 and the apparent monotonic response when survival and dissolved Cu concentrations are compared, total recoverable Cu concentrations are likely a better measure of exposure than dissolved Cu concentrations and more accurately correlate with organism response.

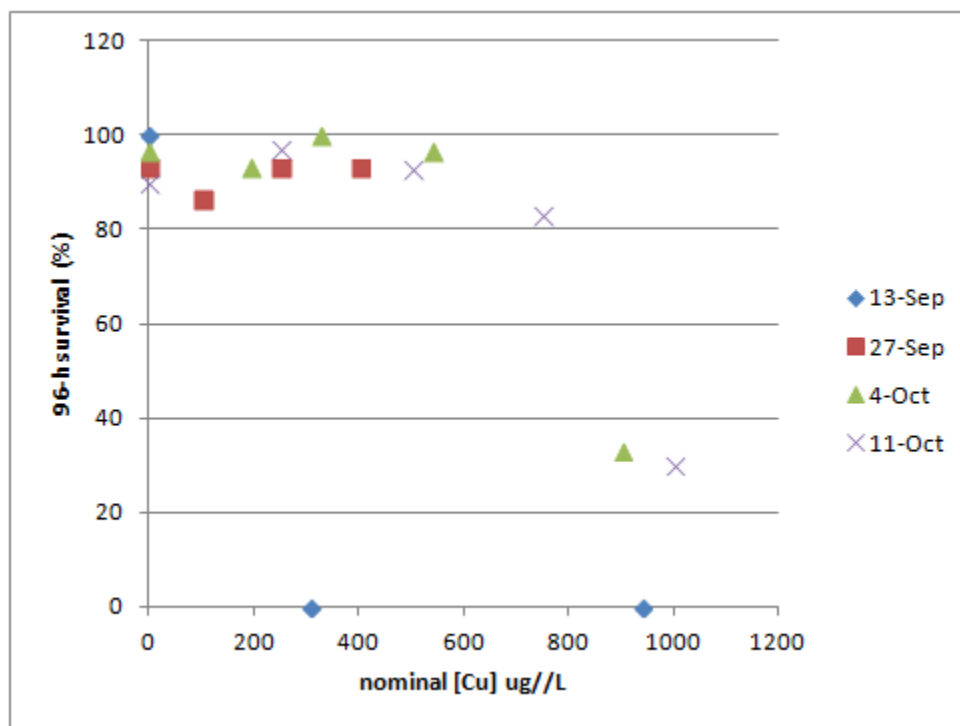


Figure 2 Results of Cu range-finding tests

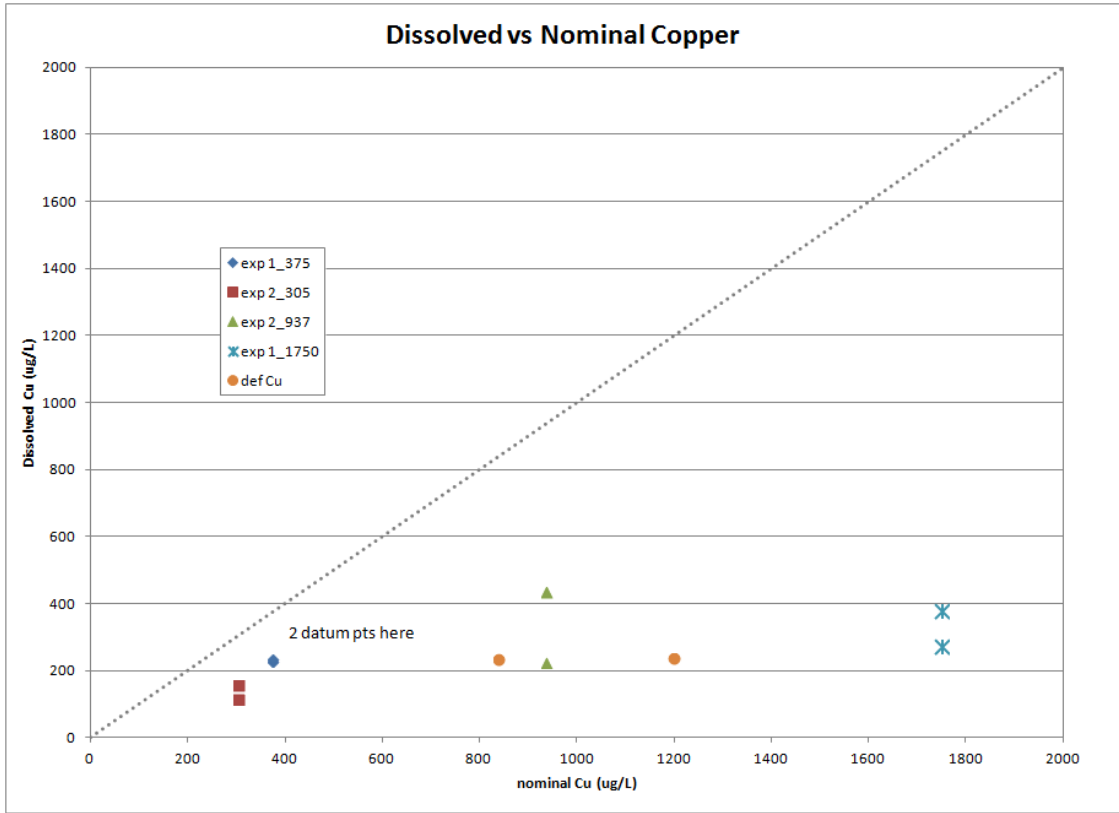


Figure 3. Dissolved Cu concentrations compared to nominal concentrations

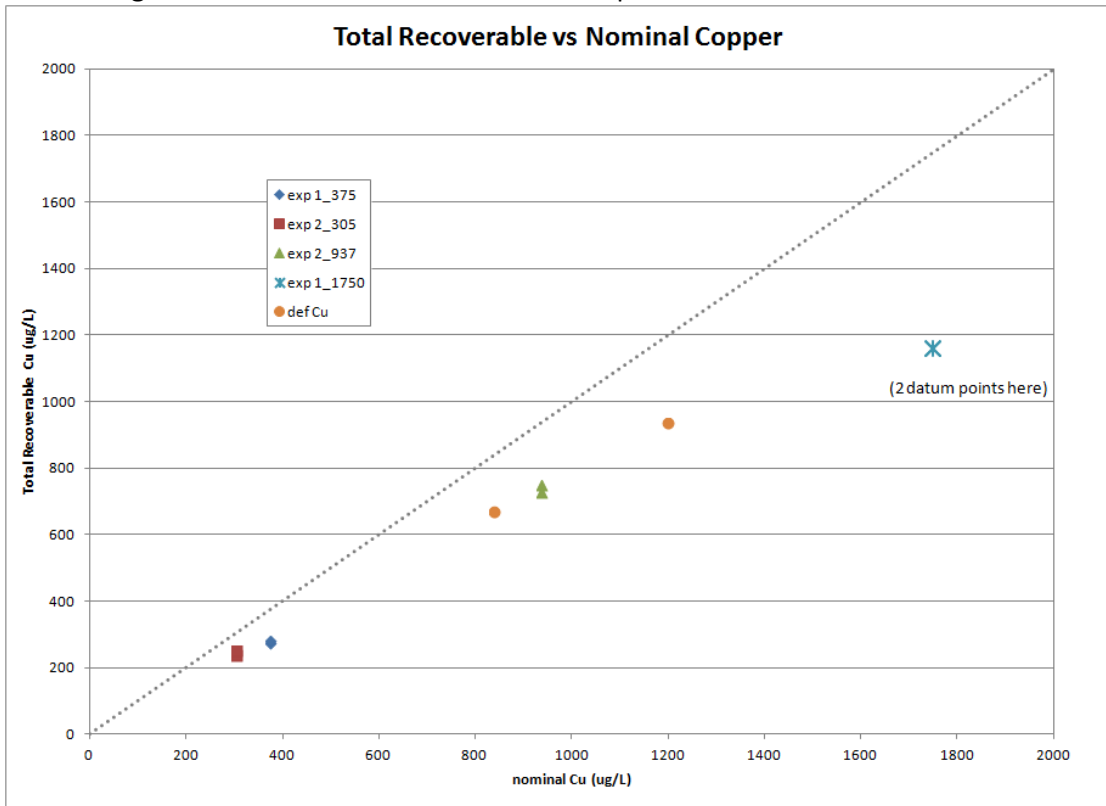


Figure 4. Total recoverable Cu concentrations compared to nominal concentrations