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RS33/RS65 – Hydrometallurgical Residue Characterization and Water Quality Model – NorthMet Project - DRAFT

Report Prepared for
PolyMet Mining Corp.

Report Prepared by



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Executive Summary

The processing and recovery of NorthMet Project ore to recover commodity metals will involve conventional flotation to produce a sulfide concentrate followed by hydrometallurgical treatment of the concentrate to recovery commodity metals. The hydrometallurgical process will produce five residues:

- Leach residue (silicates);
- Gypsum residue (calcium sulfate);
- Raffinate neutralization residue (calcium sulfate);
- Fe/Al residue (oxide); and
- Mg residue (oxide).

For final disposal, all residues will be mixed to create a single “combined residue” product which will be placed in lined disposal cells.

Samples of the residues were obtained by pilot testing and subjected to mineralogical and chemical tests to understand the composition and leaching characteristics of the residues. Except for the leach residue which is material remaining after initial leaching of the mineral concentrate, the residues are composed dominantly of calcium sulfate (mainly gypsum). The leach residue contains natrojarosite. The combined residue is also composed mainly of gypsum but natrojarosite will also be a component of this residue.

Except for the Mg residue, the individual residues were acidic to varying degrees. The combined residues were non-acidic in the time frame of the tests due to buffering minerals in the Mg residue but the presence of natrojarosite indicated that the combined residues could be acidic at some time in the future. To ensure that the combined residues do not become acidic resulting in accelerated leaching of metals held in the oxide components, additional base material (e.g. lime or limestone) will be added to offset the acid potential of the natrojarosite.

Results of the EPA1311 test shows that the residues are not classified as hazardous.

A variety of leaching tests have shown that leachate chemistry reached chemical equilibrium with the solids, and that the initial leach produced the highest concentrations of most parameters. The initial contact solution chemistry from the combined residues was used to indicate the chemistry of pore water in the hydrometallurgical residue disposal cells both during operation and at closure.

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1 Introduction

1.1 Background

PolyMet Mining Inc. (PolyMet) is proposing to develop the NorthMet Project (Dunka Road Project of US Steel) near Babbitt, Minnesota. As a part of the Minnesota Department of Natural Resources (MDNR) “Permit to Mine” process a complete “mine waste characterization” is required (Minnesota Rules Chapter 6132.1000).

The processing and recovery of NorthMet Project ore to recover commodity metals will involve conventional flotation to produce a sulfide concentrate followed by hydrometallurgical treatment of the concentrate to recovery commodity metals.

The residues from hydrometallurgical treatment will be disposed as a single combined residue. As there are indications (but no certainty) that a market can be found for the gypsum residue, this report focuses on the disposal of combination of all residues with gypsum residue as described in the Detailed Project Description (PolyMet 2007) but with reference to testwork results for combined residue without gypsum.

All residues will be disposed in lined cells. Design concepts for the cells are provided in RS28T (Barr 2007).

1.2 Objective

The objectives of the characterization studies were to evaluate the mineralogical and chemical properties of the residues and predict the chemistry of pore waters in the residues. This information is an input into the prediction of overall water quality at the hydrometallurgical residue disposal cells.

1.3 Design and Consultation Process

The characterization plan for the residues was developed in consultation with the MDNR (SRK 2005) (Appendix A). The plan covers characterization of both tailings from the flotation process and the hydrometallurgical residues from recovery of commodity metals. Characterization of the hydrometallurgical residues was started in February 2006 following generation of the residues from pilot plant testing between August 29 and October 11, 2005 and agreement on the characterization methodologies with MDNR.

1.4 Structure of Report

This report combines results of two studies. RS33 provides results of characterization of the hydrometallurgical residues whereas RS65 is the prediction of pore water chemistry.

The structure of the RS65 report, which was a combination of RS33 and RS65 was agreed with the MDNR. The final version of the report outline was transmitted to the MDNR on April 26, 2006. The agreed outline has been followed. If any sections are redundant, the section heading is shown with a brief note to explain why the section is no longer relevant.

2 Water Chemistry Prediction Methods

2.1 Theoretical Method

The theoretical method considers the rate at which components of the residues might dissolve and their final solubility. This approach is potentially applicable to the hydrometallurgical residues because they are composed primarily of discrete fully oxidized mineral phases. Solution chemistry in the pores is expected to be controlled mainly by dissolution of these phases possibly with formation of new minerals. Oxidation reactions are not expected to be significant because the residues are the product of a strongly oxidizing process.

Numerous computer modeling programs are available to predict solubility of minerals including MINTEQA2 (e.g. Allison et al 1991) and PHREEQC (Parkhurst and Appelo 1999). Other programs couple solubility, reaction paths and reaction rates (e.g. React Bethke 2005). The main limitations of these programs are thermodynamic databases and the rate expressions for mineral reactivity. For common minerals, solubility constants are well-established but for trace elements, the solubility of co-precipitated phases are site-specific and not provided in the database, which limits the value of the theoretical method as a standalone approach. However, it can be combined with the empirical approach, as described below.

2.2 Analog and Empirical Methods

2.2.1 Analog

The analog method involves direct prediction of the dissolution of residues by comparison with similar hydrometallurgical processes involving pressure oxidation of copper sulfide concentrates. Similar processes include the CESL Copper Process (TeckCominco, Undated) which involves moderate pressure oxidation of copper concentrates using chloride as a catalyst and production of residues containing leach residue, iron oxide, elemental sulfur and gypsum, and Outokumpu's HydroCopper™ process (Outokumpu 2006) which also produces iron oxide and elemental sulfur but no gypsum. These are comparable to the NorthMet process, which will result in production of jarosite as a product of sulfide oxidation and gypsum due to acid neutralization. The differences in the processes obviously limit the direct comparison of the performance of residues.

2.2.2 Empirical

The empirical method involves the use of testwork to simulate the leaching behavior of residues. The limitation of this approach is usually that the ratio of leachate to solid in testwork is often much higher than under field conditions due to the practical need to produce enough water for analysis. The results can be scaled up to field conditions by assuming that the concentration will increase in proportion to the decrease in liquid to solid ratio. Such scale-up calculations can produce very high concentrations that are not realistic.

2.3 Method Selected

The selected method is primarily empirical (i.e. based on testwork) but with evaluation of data using thermodynamic considerations, if practical, to place constraints on concentrations indicated by scale-up of laboratory results. The steps used to generate water quality predictions are therefore:

- Evaluation of overall leachate chemistry using the SpecE8 module of the Geochemist's Workbench (Bethke 2005) to determine if major elements are near chemical saturation.
- Comparison of trace metal concentrations with pH to determine if concentrations are constrained by pH.
- Scale-up of metal concentrations to reflect under-saturation indicated by SpecE8 and the pH relationships.
- Development of final predictions for pore water chemistry.

3 Program Design

3.1 Process Background

Hydrometallurgical processing will result in production of the following residues composed dominantly of the indicated minerals or mineral groups:

- Leach residue (silicates);
- Gypsum residue (calcium sulfate);
- Raffinate neutralization residue (calcium sulfate);
- Fe/Al (Iron/Aluminum) residue (oxide); and
- Mg residue (oxide).

The process flow sheets are provided in Appendix A. The pilot plant run that generated the residues for testing had a separate Fe/Al Removal Stage. Subsequent refinement of the flowsheet has combined this stage with Raffinate Neutralization which means that the raffinate neutralization residue and the Fe/Al residue will be combined in the full scale plant and are generally referred to singularly as raffinate neutralization residue.

3.2 Geochemical Background

The residues are mainly chemical products in which the original concentrate components are oxidized to sulfates and hydroxides, and dissolved.

- Sulfur from the sulfide minerals is oxidized to jarosite and soluble sulfate. The latter is precipitated as calcium sulfate through the neutralization process;
- Iron released from sulfide and silicate minerals is precipitated as hydroxide; and
- Magnesium and aluminum released from silicate minerals are precipitated as hydroxides.

Copper is recovered in the process by electro-winning to produce copper anodes. Nickel, cobalt and zinc are recovered as a mixed hydroxide product. Platinum group metals and gold are recovered as concentrate product.

Incomplete dissolution of the silicate and sulfide minerals results in the generation of a fourth residue; a mineralogical leach residue.

Because these products represent near-complete dissolution, oxidation and neutralization of the concentrate, the chemistry of contact waters is expected to be controlled mainly by the simple

dissolution of the compounds. Oxidation of residual sulfide minerals may occur from the leach residue but this effect is expected to be minor.

Overall, the process of dissolution of neutralization products is unrelated to the composition of the ore and is not expected to vary in the long term except by depletion.

3.3 Data Requirements

The primary requirements for the test program were therefore:

- Mineralogical characterization of residues to provide a basis for understanding the dissolution of the residues; and
- Dissolution tests to investigate the solubility of the minerals.

3.4 Overall Program Design

The overall program design included the following components:

- Production of individual and combined residues using a pilot plant (see RS32 Part III, Barr 2006).
- Mineralogical characterization of residues.
- Determination of solids elemental characteristics.
- Performance of various types of leaching experiments at different solid to liquid ratios to determine the dissolution behavior of the residues.

4 Sampling and Analytical Methods

4.1 Metallurgical Program

4.1.1 Ore Composite Preparation

Preparation of the ore composite is described in ER03 (PolyMet, in preparation).

4.1.2 Generation of Residue Samples

Sulfide concentrates were prepared by a pilot flotation plant in which the copper sulfate was evaluated as a reagent to improve flotation of sulfide minerals. Two concentrates were prepared in the pilot plant. One concentrate was prepared with the use of copper sulfate, and a second was prepared without the use of copper sulfate. Both residues were subsequently leached in the hydrometallurgical process. PolyMet has since decided that copper sulfate will be used. Therefore, it was agreed (in consultation with MDNR, SRK 2005) that characterization testwork would be performed on the residues generated by leaching of the concentrate that was produced with copper sulfate. The exception is the leach residue for which both residue samples are being tested.

Residues generated during the pilot plant run at SGS Lakefield were collected under supervision of Barr Engineering and shipped to Canadian Environmental and Metallurgical Inc (CEMI) in Burnaby, British Columbia, Canada for testing. SRK Consulting is responsible for supervision of the testwork described in this report.

Testing is proceeding on individual samples of each type of residue and combined residues. Residue combinations are being tested with and without the gypsum residue to allow for the option of recovery of a separate gypsum product. The proportions by wet weight of each residue in the combined samples are provided in Table 4-1. The combined residue containing all residues was received directly from the pilot plant (SGS Lakefield). The combined residue without gypsum was prepared from the component residues using the same weight proportions less the gypsum amount.

Table 4-1: Proportions (by Wet Weight) for Combined Residues

Residue	Actual Pilot Plant Observation		Projected for Commercial Scale
	All	Without Gypsum	All
Leach Residue	27%	45%	41%
Gypsum Residue	40%	0%	33%
Raffinate Residue	18%	31%	20%
Fe/Al Residue	6%	10%	NA, combined w/Raffinate
Mg Residue	8%	14%	6%

Subsequent optimization and modeling (MetSim Version U3) of the hydrometallurgical process by Bateman indicated that the proportion of the residues will be somewhat different under full scale production (see Table 4-1). Specifically, the pilot plant run that generated the residues for testing had a separate Fe/Al Removal Stage. The commercial scale flowsheet has combined this stage with Raffinate Neutralization which means that the raffinate neutralization residue and the Fe/Al residue will be combined in the full scale plant. This difference will affect the overall mineralogical make-up of the residues but will not affect overall leaching performance. Further discussion is provided in Section 6.4.

4.2 Dissolution Testwork

4.2.1 Mineralogy

Mineralogical characterization included:

- Optical Analysis on feed, product and mineral wastes (Leach Residue). Other residues were not examined optically because they are precipitates; and
- X-Ray Diffraction on all samples to determine crystalline compounds.

Residues were not examined sub-optically due to the nature of the materials.

4.2.2 Analytical Methods

Solids Characterization

A split of each sample was submitted for:

- Sulfur forms (total S, S as sulfate).
- Paste pH.
- Neutralization potential and carbonate.
- 50 elements (mostly metals by ICP scan following aqua regia (nitric and hydrochloric acids) digestion).
- Whole rock oxides. This provides total concentrations of major elements.

Leachate Extraction Tests

All samples were submitted for regulatory leach tests (EPA 1311; EPA 1312) to provide data for waste classification purposes.

A third leach procedure was used as the first step of the sequential shake flask leach procedure described below. This procedure, developed by Price (1997) for the British Columbia (Canada) Ministry of Energy Mines and Petroleum Resources, uses a lower leach ratio (1:3) to improve

detection of low levels of metals. The lixiviant was deionized water, which typically has a pH between 5 and 6. The leachate pH was not fixed as in the TCLP and SPLP methods. The extraction was performed by shaking in a glass or plastic container for 24 hours, after which the leachate was extracted and analyzed.

Extraction test results are provided in Appendix B.

Sequential Shake Flask

All samples were tested using a sequential leach procedure consisting of weekly repetition of the leach procedure developed by Price (1997) in which the solid to liquid ratio is 1:3. The procedure involved weekly leaching of roughly 300 g of solids in a plastic bottle. The leaching step consisted of addition of deionized water and agitation for 24 hours. The leachate was then decanted for analysis.

This procedure was requested by the MDNR to provide a more aggressive evaluation of residue dissolution than was perceived to occur in humidity cells.

Leachates were analyzed using the following schedule:

- Weekly – pH, oxidation-reduction potential, conductivity.
- Bi-weekly – Acidity, alkalinity, inorganic C, hardness, anions (F, Cl, SO₄).
- Four weekly (weeks 0, 4, 8, 12, etc) – Low level element scan using ICP-MS.
- Four weekly (weeks 2, 6, 10, 14, etc) – General element scan using ICP-OES.

Humidity Cell

Samples were tested in ASTM-style humidity cells in the tailings configuration. Details of the procedure are provided in Appendix A. The solid to liquid ratio is 1:0.5 (1 kg of solids leached with 500 mL of deionized water). Leachates were analyzed using the same schedule as the sequential shake flasks.

MDNR Reactor

Samples were also tested in small (75 g) MDNR Reactors for which the solid to liquid ratio is 1:2.7 (75 g of solids leached with 200 mL of deionized water). Details are provided in Appendix A. Leachates were analyzed using the same schedule as the sequential shake flasks.

Kinetic Testwork Duration and Data Management

The dissolution tests were started in early February 2006. Up to 38 weeks of data are available depending on the parameter. Tests are ongoing. Graphs illustrating concentrations obtained are

provided in Appendix C. This report is based on data collected and checked for quality to the end of October 2006.

Because different reporting limits were used for different cycles and different parameters, the following rules were used to plot data and allow trends to be apparent:

- If the parameter was not determined, the result is not plotted. This is the case for alkalinity and acidity which are only determined if the pH is above or below (respectively) specified values.
- If the result is undergoing quality control re-check, the result is not plotted.
- If the parameter is determined by the same method for each analysis, values below the reporting limit values are plotted as 50% of the reporting limit.
- Because the reporting limits for ICP-MS are below the reporting limit for ICP-ES:
 - If the result was determined by ICP-MS and was below the reporting limit, the value on the graph is 50% of the reporting limit. If the value is at or above the reporting limit, the value is plotted.
 - If the result was determined by ICP-ES and was determined to be below the reporting limit, no value is plotted.
 - If the result was determined by ICP-ES and was determined to be above the reporting limit, the value is plotted.
 - These rules can result in four cycles between plotted results if the parameter is not detected by ICP-ES (e.g. molybdenum in shake flask leachates).

Occasionally, “sawtooth” trends are apparent in which values alternate between high and low for the ICP-ES and ICP-MS analyses. This results from analytical “noise” around the ICP-ES reporting limit when reported values are slightly above the reporting limit. Aluminum is a particular example that commonly shows reported values above the ICP-ES reporting limit of 0.05 mg/L.

Many graphs are plotted on logarithmic axes to allow data spanning a wide range of concentrations to be compared.

4.2.3 Quality Assurance/Quality Control

In addition to Quality Assurance/Quality Control (QA/QC) of leachate chemistry performed by the analytical laboratory, QA/QC on the dissolution test procedure and overall leachate chemistry included blanks, duplicates, leachate ion balances and visual trend analysis to identify severe outliers. These measures were designed mainly in conjunction with the waste rock characterization plan, the results of which are described in RS42.

Quality assurance review for the waste rock and tailings programs identified two specific issues with respect to pH measurements and antimony leaching that affected the subsequent interpretation of the results. These issues also applied to the residue program and are discussed in the following paragraphs.

Trend analysis of pH measurements indicated a “sawtooth” trend in which values alternated between higher and lower values every other week. The reason for the pattern was that pH measurements were performed on filtered and unfiltered leachates on alternate weeks depending on whether samples were being collected for metals analysis. This was consistent with the analytical method. Because vacuum filtration potentially causes weakly buffered leachates to respond to changes in pressure by taking up or releasing carbon dioxide, determination of pH of filtered leachates was discontinued when the concern was identified. For results prior to this point in the test program, pH measurements on filtered leachates were discarded. Where pH results were needed for interpretation of other chemical parameters, the two nearby results were averaged (i.e. assuming the results could be interpolated linearly).

Review of antimony data indicated that test apparatus components of humidity cell tests constructed from polyvinyl chloride (PVC) were leaching antimony due to the use of antimony oxide in manufacturing. Antimony results from humidity cell results were therefore discarded.

4.2.4 Interpretation Methods

Results were interpreted using three methods:

- Leachate chemistry trends were examined as a function of time.
- All leachate results were combined and evaluated with respect to pH as the primary control on concentrations.
- Selected leachate results were input into a thermodynamic chemical equilibrium model to evaluate chemical saturation.

5 Results

5.1 Solids Characteristics

Table 5-1 provides quantitative mineralogy determined by Rietveld x-ray diffraction. Results are expressed as weight percentage of crystalline phases. If amorphous phases are present, the quantities of the indicated crystalline phases will be lower than indicated.

Table 5-2 provides elemental composition of the residues.

Table 5-3 provides sulfur forms from analysis and mineralogy, neutralization potential, carbonate analyses and acid-base accounts. The distribution of sulfate indicated by mineralogy was calculated as follows:

- The proportions indicated by mineralogy for natrojarosite, gypsum and bassanite were used to calculate the quantity of sulfate indicated by mineralogy using the formula weights of these minerals.
- The total sulfate indicated by mineralogy was calculated and compared to the analyzed sulfate. As shown in Table 5-3, there is a strong correspondence between the analytical and mineralogical sulfate amounts.
- The proportion of the analytical sulfate as gypsum and bassanite was calculated based on the proportion of these minerals indicated by mineralogy.

Acid potential (AP) in Table 5-3 was calculated based on sulfur not occurring as calcium sulfate based on the assumption that sulfur as natrojarosite is the source of acid. The factor for converting sulfur as natrojarosite to AP is 23.44 compared to 31.25 for sulfur as iron sulfide.

Table 5-1: Mineralogy of Residues

Mineral	Unit	Leach, no CuSO ₄	Leach, with CuSO ₄	Gypsum	Raffinate Neutralization	Fe/Al	Mg	Combined	Combined no Gypsum
Quartz	%	2.1	3	0.2	0.3	0.3	0.2	2.2	3.4
Plagioclase	%	25.6	5.8						2.2
Gypsum	%	8.1	6.9	99.8	96.3	98.9	76.8	73.4	53.3
Calcite	%							2.9	
Bassanite	%				3.4				
Siderite	%							1.2	
Actinolite	%	1.3							
Talc	%	4.3	3.9					3.2	3.2
Natrojarosite	%	34	63					9.6	29.3
Hematite	%	24.6	17.4					6	8.6
Goethite	%					0.8			
Butlerite	%							1.5	
Brucite	%						22.2		
Halite	%						0.8		

Leach Residues

The leach residues remain from the concentrate following pressure leaching. It was expected that this product would consist of both secondary minerals and resistant primary minerals (silicates).

Leach residues from processing of concentrates produced by sulfide flotation with and without copper sulfate were tested. The XRD results implied differences in the residual silicate component of the residues. The concentrate produced without copper sulfate had 26% residual plagioclase compared to 6% for the concentrate produced with copper sulfate. The oxidation products produced by the process are shown as mainly natrojarosite, which was also confirmed by the sodium content of the samples (Table 5-2). Hematite (iron oxide) and talc (basic magnesium silicate) were also present.

The residual metal content of the leach residues was significantly lower when the feed concentrate was generated using copper sulfate. Nickel, cobalt and zinc were an order-of-magnitude lower.

As expected, the leach residues were acidic and contained residual acidity shown by negative NP. In addition, the presence of natrojarosite resulted in higher acid potential than the other residues (111 and 199 kg CaCO₃/t).

Table 5-2: Elemental Composition of Residues

Parameter	Unit	Leach, no CuSO ₄	Leach, with CuSO ₄	Gypsum	Raffinate Neutralization	Fe/Al	Mg	Combined	Combined no Gypsum
Ag	ppm	11.05	23.8	0.23	0.9	0.33	0.12	4.93	11.05
Al	%	2.82	1.97	0.02	0.02	0.46	0.03	0.54	1.01
As	ppm	35.4	56.3	3	6	15	5	110	34
B	ppm	<10	<10	<10	<10	<10	<10	<10	<10
Ba	ppm	40	30	<10	<10	<10	<10	10	20
Be	ppm	0.05	<0.05	<0.05	<0.05	0.28	<0.05	<0.05	<0.05
Bi	ppm	4.89	6.84	0.5	0.8	0.81	0.04	1.94	3.53
Ca	%	1.88	1.41	14.5	17.1	18.8	15.1	16.35	11.3
Cd	ppm	0.66	0.19	0.22	0.73	0.48	0.44	0.29	0.23
Ce	ppm	8.21	5.45	1.71	0.6	0.85	1.6	2.35	2.93
Co	ppm	133.5	17.9	5.1	9.3	105.5	13.6	6.9	23.6
Cr	ppm	112	154	8	11	457	5	59	116
Cs	ppm	0.1	<0.05	<0.05	<0.05	0.09	<0.05	<0.05	<0.05
Cu	ppm	7380	1280	184.5	22.5	2960	26.9	519	945
Fe	%	17.3	26.9	0.05	0.08	1.88	0.05	5.21	11.75
Ga	ppm	6.47	5.06	2.54	0.07	0.57	0.09	1.22	2.43
Ge	ppm	0.41	0.47	<0.05	<0.05	0.08	<0.05	0.11	0.22
Hf	ppm	0.12	0.06	<0.02	<0.02	0.12	<0.02	0.04	0.04
Hg	ppm	0.04	0.02	0.12	0.35	0.07	0.05	0.11	0.03
In	ppm	0.3	0.355	23.3	0.011	1.01	0.083	0.164	0.28
K	%	0.11	0.1	<0.01	<0.01	0.01	0.01	0.02	0.05
La	ppm	3.6	2.5	0.7	0.5	0.5	2.3	1.1	1.6
Li	ppm	1	0.6	0.1	0.1	0.5	0.5	0.2	0.2
Mg	%	0.28	0.25	0.01	0.01	0.14	9.44	0.39	1.47
Mn	ppm	35	<5	<5	5	23	83	<5	<5
Mo	ppm	21.8	28.4	0.47	0.85	42.3	0.38	9.35	18.9
Na	%	1.28	3	0.02	0.04	0.13	1.04	0.58	1.62
Nb	ppm	0.18	0.21	<0.05	0.06	0.46	0.06	0.11	0.17
Ni	ppm	3270	410	99.9	192.5	2710	1230	260	674
P	ppm	290	110	<10	20	160	40	70	90
Pb	ppm	48.5	56.1	8.3	86.1	20.4	12.5	133.5	32
Rb	ppm	1.7	1	0.2	0.3	0.5	0.3	0.4	0.7
Re	ppm	0.012	0.002	0.015	0.022	0.013	<0.001	0.009	0.009
S	%	5.47	9.22	>10.0	>10.0	>10.0	>10.0	>10.0	>10.0
Sb	ppm	1.22	1.81	0.18	1.25	1.47	0.4	3.99	1.14
Sc	ppm	1.3	0.8	0.4	0.4	4.4	0.5	0.8	1.1
Se	ppm	49.8	73.4	0.3	0.4	0.6	0.5	20.4	39.2
Sn	ppm	4.8	6.1	0.2	0.5	1.6	<0.2	2	3.2
Sr	ppm	73.1	63.9	60.6	106	83.7	86.5	77.3	85.2
Ta	ppm	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Te	ppm	2.01	2.91	0.01	0.07	0.09	<0.01	0.83	1.48
Th	ppm	1.1	0.9	<0.2	<0.2	0.3	<0.2	0.3	0.4
Ti	%	0.066	0.042	<0.005	<0.005	0.007	<0.005	0.012	0.019
Tl	ppm	0.1	0.34	0.03	0.03	0.24	0.02	0.06	0.19
U	ppm	0.06	<0.05	<0.05	<0.05	0.55	0.1	0.06	0.09
V	ppm	39	41	1	1	16	1	11	21
W	ppm	0.57	0.49	0.09	0.18	1.12	<0.05	0.26	0.45
Y	ppm	3.88	3.29	1.28	1.14	1.06	7.64	2	2.86
Zn	ppm	171	15	30	98	164	23	32	36
Zr	ppm	<0.5	0.7	1.5	4.2	1.2	<0.5	9.8	1.9

Table 5-3: Acid-Base Accounting Results

Parameter	Unit	Leach, no CuSO ₄	Leach, with CuSO ₄	Gypsum	Raffinate Neutralization	Fe/Al	Mg	Combined	Combined no Gypsum
Paste pH	-	2.6	3.3	3.8	5.4	4.6	9.6	-	9.4
Sulphur Forms									
Total S	%	6.24	9.8	19.55	18.95	17.9	14.4	16.5	13.65
S as SO ₄	%, S	6.1	9.3	18.35	18.55	17.3 5	13.6 5	15.9	13.5
Calculated Sulfur Forms From XRD Mineralogy									
S as Gypsum	%, S	1.51	1.28	18.55	17.90	18.3 8	14.2 7	13.64	9.90
S as Natrojarsoite	%, S	4.49	8.32	0.00	0.00	0.00	0.00	1.27	3.87
Total SO ₄	%, S	5.99	9.61	18.55	17.90	18.3 8	14.2 7	14.91	13.78
Calculated Sulfur Not Present as Calcium Sulfate									
Non-Gypsum	%, S	4.73	8.52	1.00	1.05	-0.48	0.13	2.86	3.75
Neutralization Potential									
Fizz Rating	-	1	1	1	1	1	3	1	2
Neutralization Potential (NP)	kg CaCO ₃ /t	-17	-4	0	0	-10	371	10	51
CO ₂	%	-0.2	-0.2	-0.2	-0.2	-0.2	1.8	-0.2	0.3
C	kgCaCO ₃ /t	2	2	2	2	2	40	2	7
Acid Potential (AP)	kg CaCO ₃ /t	111	199	23	25	-11	3	67	88
Acid-Base Accounting									
NP-AP	kg CaCO ₃ /t	-164	-271	-38	-13	-27	348	-51	-72
NP/AP	-	-	-	0.0	0.0	-	124	0.1	0.6

Gypsum Residue

Gypsum residue is the first hydrometallurgical precipitation product. It is produced by limestone addition to the leach solution following recovery of platinum group metals and prior to copper recovery.

The elevated calcium and sulfate content of the gypsum residue sample confirmed that it was dominantly hydrated calcium sulfate. XRD showed that it was 99.8% gypsum.

The metal content of this residue was very low. Acid-base accounting indicated that the dominant sulfur form was sulfate. About 1.2% of the sulfur was not accounted for by sulfate analysis, but XRD failed to recognize any other sulfur minerals. It is likely therefore that the difference reflects analytical uncertainties rather than unknown mineral content.

Raffinate Neutralization Residue

This residue is formed by an intermediate neutralization step between copper removal and precipitation of iron and aluminum.

Raffinate neutralization residue was identified as nearly entirely calcium sulfate (gypsum with minor bassanite) by XRD, which was confirmed by the dominance of calcium and sulfate in the sample. Like the gypsum residue, the metal content of this residue was very low and the sulfur forms analysis was consistent with the dominance of sulfate.

Fe/Al Residue

The Fe/Al residue is formed by two limestone addition steps prior to cobalt, nickel and zinc hydroxide recovery. Note that the full scale plant design combines Fe/Al removal with raffinate neutralization which means that the Fe/Al residue will be combined with the raffinate neutralization residue.

Like the raffinate neutralization residue, the Fe/Al residue was mostly gypsum with some iron and aluminum. Goethite was detected by XRD and iron and aluminum were both an order of magnitude higher than the other two dominantly calcium sulfate residues. The Fe/Al residue contained higher concentrations of copper and nickel compared to the gypsum and raffinate neutralization residues.

Magnesium Residue

The magnesium residue is formed following recovery of the mixed hydroxide product and is the result of final addition of lime to the process solutions. The magnesium originates from the original concentrate leaching step (dissolution of olivine), and addition of magnesium oxide to precipitate the mixed hydroxide product.

The crystalline component of this residue was dominated by gypsum as shown by the elevated sulfate and calcium. The XRD scan showed that brucite (magnesium hydroxide) was present and this was supported by the magnesium content of the sample. The sample showed a strong fizz reaction to hydrochloric acid and carbonate content was equivalent to 4% calcium carbonate (40 kg CaCO₃/t). This suggests that the hydroxide was partially converted to carbonate by atmospheric reaction.

Neutralization potential was high at 371 kg CaCO₃/t compared to the other residues; probably reflecting the presence of amorphous magnesium and possibly calcium hydroxide. This residue was alkaline (pH 9.6) and consistent with the hydroxide content.

The dominant trace metal in this residue was nickel (1230 mg/kg).

Combined Residues including Gypsum Residue

The largest component of the combined residue was the gypsum residue, followed by leach residue, raffinate residue, Mg residue and Fe/Al residue. As a result the material was dominantly calcium sulfate from the gypsum residue. The XRD result also showed the presence of calcite and siderite, presumably due to the magnesium residue.

The overall acid-base account for this sample indicated AP of 67 kg CaCO₃/t and NP of 10 kg CaCO₃/t. Because carbonate content was relatively low compared to NP, most NP was present as hydroxide from the magnesium residue. As a result, the residue was predicted to be potentially acid generating (NP/AP of 0.1). The acid generation potential is produced by natrojarosite which yields acidic leachate when dissolved. This process does not require an oxidant.

Combined Residues without Gypsum Residue

Although this combination did not include the gypsum residue, gypsum remained a significant component from the raffinate neutralization, Fe/Al residue and Mg residues. The overall characteristics of this material are similar to the combination of all residues though both AP and NP were higher in this residue due to the higher proportions of leach and magnesium residues respectively. The NP/AP of the residue was calculated to be 0.6.

5.2 Description of Leachate Chemistry

5.2.1 EPA 1311

Results for EPA 1311 (TCLP) are provided in Appendix B. Concentrations for all parameters were below regulated limits. None of the residues (individually or combined or combined without the gypsum residue) are classified as hazardous wastes.

5.2.2 EPA 1312

Results for EPA 1312 (SPLP) are provided in Appendix B. The use of weakly buffered acidic solution resulted in a range of final leachate pHs that reflect the stage of neutralization in the process. The leach residues had the lowest pH (2.79 and 3.38), followed by gypsum (4.1), raffinate neutralization (4.9), Fe/Al (5.0) and magnesium (9.7). The combined residues had pHs of 9.2 (all residues) and 9.4 (no gypsum residue). The variation in pH also resulted in different metal concentrations in solution. The leach residues had the highest metal concentrations. The two gypsum dominated residues had much lower metal concentrations (despite the lower pH). The higher metal content of the Fe/Al residue resulted in higher leachate metal concentrations. The three non-acidic leachates had low metal concentrations.

5.2.3 Price (1997) Method

Results for Price (1997) method are provided in Appendix B. This method uses deionized water as its extractant and a low liquid to solid ratio (3:1) compared to SPLP and TCLP which both use 20:1. The Price (1997) method is closely related to the SPLP method which uses deionized water weakly buffered by sulfuric and nitric acids. Both of these methods are different from the TCLP method which uses acetic acid to buffer pH.

As a result of the low liquid to solid ratio in the Price (1997) method, leachate pHs were lower than for the SPLP for the more acidic residues. The two leach residues had pHs of 2.1 and 2.6, followed by gypsum (3.3) and raffinate neutralization (4.2). For the three non-acidic residues, the pH was slightly lower with the Price (1997) method.

Differences in metal concentrations between the SPLP and Price (1997) methods appear to reflect the different liquid to solid ratios. Both copper and nickel concentrations were 4 to 7 times higher in the Price (1997) method leachates for the leach, gypsum, raffinate neutralization and Fe/Al residues. Concentrations of these elements in leachates from the three non-acidic residues were relatively low which obscured differences between the leachates.

5.3 Description of Kinetic Test Leachate Chemistry

5.3.1 Sequential Shake Flasks

Sequential shake flask charts are provided in Appendix C.1. Data are provided on a CD included in the report pocket.

Samples of leach residues, gypsum residue, raffinate neutralization residue and Fe/Al residue consistently produced acidic leachate but pH steadily increased for all residues except for the leach residue from processing of concentrate produced using copper sulfate. The raffinate neutralization residue showed erratic increase in pH reaching high values above 7. Leachates were dominated by calcium and sulfate. Sulfate concentrations were lowest for the leach residues (60 and 200 mg/L in most recent samples) but stable at about 1600 mg/L for the other acidic residues. Trends for other parameters included:

- Aluminum concentrations were greatest for the leach residues and slowly increased up to 3.4 mg/L for the residue produced from concentrate using copper sulfate. Other tests showed declining and low concentrations.
- Cobalt, copper, nickel and zinc concentrations were greatest for the Fe/Al residue as the test proceeded (0.003 mg Co/L, 1.4 mg Cu/L, 0.2 mg Ni/L and 0.02 mg Zn/L in most recent leachate) and steadily declined in all cases.
- Iron concentrations were greatest but declining for the gypsum residue (1.7 mg/L in a recent leachate).
- Lead concentrations were greatest and declining for the raffinate neutralization residue (0.01 mg/L in latest sample).
- Sodium concentrations were greatest for the leach residues and slightly increasing (7 mg/L in recent leachate).

Magnesium residues showed declining pH from 9.6 initially to 6.4 in recent leachates. Leachates were dominated by sulfate concentrations at higher levels than other residues (2,500 to 7,900 mg/L)

and magnesium rather than calcium as the dominant cation. Sodium and chloride concentrations were initially elevated (both above 1000 mg/L) then near to 1 mg/L. Concentrations of metal ions were very low and stable or declining. The exception was boron, which showed increasing concentrations reaching 0.16 mg/L in recent leachates.

Combined residues showed stable or very slightly declining pH reaching about 7. Leachate chemistry was dominated by calcium and sulfate at narrowly constrained stable concentrations. Alkalinity leaching declined but appeared to stabilize at about 30 mg CaCO₃/L. Major element chemistry reflected mixing of waters from the individual residues. Metal concentrations were generally low. The following exceptions were apparent:

- Arsenic leaching from both residues was greater than other residues and appeared to be stable. Greatest concentrations were 0.003 mg/L.
- Molybdenum leaching in these residues was greater than other residues. Maximum concentrations were 0.03 mg/L in both combined residues, but these concentrations declined. The combined residue without gypsum showed higher concentrations than the combined residue.
- Selenium concentrations were also greatest for these residues and showed a stable trend following decreases. The combined residue without gypsum showed higher concentrations (maximum of 0.019 mg/L). Concentrations in most recent samples were 0.007 mg/L.

5.3.2 Humidity Cells

Humidity cell results charts are provided in Appendix C.2.

Samples of leach residues, gypsum residue, raffinate neutralization residue and Fe/Al residue consistently produced acidic leachate but pH steadily increased for all residues, except for the leach residue from processing of concentrate produced using copper sulfate. The raffinate neutralization residue showed the greatest increase in pH (from 3.3 to 4.6, except for one point at 6.0). Leachates were dominated by calcium and sulfate. Sulfate concentrations rapidly stabilized between 1600 and 1900 mg/L, becoming 1600 mg/L as the test proceeded. Concentrations of other parameters either remained stable or decreased, for example:

- Aluminum concentrations were greatest for the most acidic leach residues (up to 14 mg/L following an initial flush) but remained relatively stable.
- Cobalt, copper, nickel and zinc concentrations were greatest for the Fe/Al residue as the test proceeded (0.03 mg Co/L, 10 mg Cu/L, 2.8 mg Ni/L and 0.25 mg Zn/L in most recent leachate) and steadily declined in all cases.
- Iron concentrations were greatest and relatively stable for the gypsum residue (1.7 mg/L in a recent leachate).
- Lead concentrations were greatest and declining for the raffinate neutralization residue (0.05 mg/L in latest sample).

- Sodium concentrations were greatest for the leach residues and stable or slightly decreasing.

Acidity of these leachates was stable or slowly declining for the leach residues at near 100 mg CaCO₃/L, and declining at lower levels for the gypsum dominated residues.

Magnesium residues showed declining pH from 9.4 initially to 7.4. Leachates were dominated by sulfate concentrations at higher levels than other residues (5,000 to 10,000 mg/L) and magnesium rather than calcium as the dominant cation. Sodium and chloride concentrations were initially elevated (both above 1000 mg/L) then declined to less than 10 mg/L. Concentrations of metal ions were very low and stable or declining.

Combined residues showed stable or very slightly declining pH near 7 after about 20 weeks. Leachate chemistry was dominated by calcium and sulfate. Alkalinity leached at low levels (about 23 mg CaCO₃/L in recent leachates). Major element chemistry reflected mixing of waters from the individual residues. For example, chloride leaching initially followed the same trend as the magnesium residue for the combined residue without gypsum. Metal concentrations were generally low. The following exceptions were apparent:

- Arsenic leaching from both residues showed an increasing trend though at low levels. The combined residue showed the greatest concentration in any residue leachate (0.007 mg/L).
- Molybdenum leaching in these residues was greater than any other residue. Maximum concentrations were 0.14 mg/L in the combined residue, but these concentrations declined. The combined residue without gypsum showed stable concentrations between 0.03 and 0.09 mg/L.
- Selenium concentrations were also greatest for these residues and showed a slowly increasing trend for the combined residue without gypsum. Concentrations in most recent samples were 0.03 mg/L.

5.3.3 MDNR Reactors

MDNR reactor results charts are provided in Appendix C.3.

Samples of leach residues, gypsum residue, raffinate neutralization residue and Fe/Al residue consistently produced acidic leachate but pH steadily increased for all residues except for the leach residue from processing of concentrate produced using copper sulfate. Like other tests, the raffinate neutralization residue showed the greatest increase in pH (from 3.3 to 4.6). Leachates were dominated by calcium and sulfate. Like the sequential shake flask results, sulfate concentrations were lowest for the leach residues (90 and 200 mg/L in most recent samples) but stable between 1400 and 1600 mg/L for the other acidic residues. Concentrations of other parameters either remained stable or decreased, for example:

- Aluminum concentrations were greatest for the most acidic leach residues (up to 2.8 mg/L following an initial flush) but remained relatively stable.

- Cobalt, copper and nickel concentrations were greatest for the Fe/Al residue as the test proceeded (0.006 mg Co/L, 3.1 mg Cu/L, and 0.5 mg Ni/L in most recent leachate) and steadily declined in all cases.
- Zinc concentrations were comparable in Fe/Al residue and raffinate neutralization residue as the test proceeded. Zinc concentrations in most recent leachates were about 0.04 mg/L.
- Iron concentrations were similar and relatively stable after about 20 weeks for the leach residue (copper sulfate used for concentrate), gypsum residue and raffinate neutralization residues (highest concentrations of about 0.2 mg/L).
- Lead concentrations were greatest and declining for the raffinate neutralization residue (0.02 mg/L in latest sample).
- Sodium concentrations were greatest for the leach residues and stable or slightly decreasing.

Magnesium residues showed declining pH from 9.8 initially to 7.4. Leachates were dominated by sulfate concentrations generally at higher levels than other residues (up to 5,000 mg/L) and magnesium rather than calcium as the dominant cation. Sodium and chloride concentrations were initially elevated (both above 200 mg/L) then declined to near or less than 1 mg/L. Concentrations of metal ions were very low and stable or declining.

Combined residues showed stable or very slightly declining pH between 7 and 7.4 after about 9 weeks. Leachate chemistry was dominated by calcium and sulfate. Alkalinity leached at low levels (about 23 mg CaCO₃/L in recent leachates). Major element chemistry reflected mixing of waters from the individual residues. For example, chloride leaching initially followed the same trend as the magnesium residue for the combined residue without gypsum. Metal concentrations were generally low. The following exceptions were apparent:

- Arsenic leaching from both residues was greater than other residues and appeared to be stable. Greatest concentrations were 0.002 mg/L.
- Boron leaching from the combined residue showed an increasing trend reaching a maximum concentration of 0.27 mg/L.
- Molybdenum leaching in these residues was greater than any other residue at first but then decreased and showed similar concentrations to the Fe/Al Residue. Maximum concentrations were 0.06 mg/L in the residue without gypsum, but these concentrations declined.
- Selenium concentrations were also greatest for these residues and showed a stable trend with maximum concentrations of 0.008 mg/L.

6 Interpretation of Dissolution Testwork

6.1 General Interpretation of Leachate Chemistry

6.1.1 Saturation Indices

In order to interpret the testwork chemistry, selected leachate chemistry data from all tests were input into Geochemists' Workbench (Bethke 2005) to evaluate whether the leachates were in equilibrium with any of the known mineral components of the residues. As shown in the foregoing descriptions, the humidity cells yielded the highest concentrations of metals. This is consistent with the low applied liquid to solid ratio in these tests (0.5 mL/g) compared to the sequential shake flasks (3 mL/g) and MDNR Reactors (2.7 mL/g). The humidity cells are therefore most likely to show chemical saturation. Table 6-1 shows saturation indices for minerals identified by XRD and other minerals that may control solubility. Color coding shows leachates that are close to chemical saturation (green shading for $-0.5 < SI < 0.5$) and well over-saturated (amber for $SI > 0.5$). Saturation indices were calculated for the initial release and chemistry after 28 weeks following the decrease that typically occurred in the early weeks.

Leachate from leach residues appeared to be constrained by dissolution of silica and gypsum both initially and as the test proceeded. While it is likely that natrojarosite is dissolving releasing ferric iron, the low pH of the leachates means that the solubility of natrojarosite is not limited and ferric hydroxide does not precipitate. The gypsum and raffinate neutralization residues show much the same result though it appears that under initial conditions fluorite was also dissolving or forming in the raffinate neutralization residue as shown by SI above 1. Fluoride concentrations were 42 mg/L initially. In the higher pH Fe/Al residues, gypsum continued to be an important overall chemistry control, but ferric hydroxide was indicated as at saturation meaning that iron oxides were probably controlling solution chemistry. Also, the SI for tenorite was -1.2 compared to lower than -4.4 for the more acidic residues. For the magnesium residue, brucite and carbonates were near or well saturated consistent with the mineralogy of the samples. This effect was most apparent in the early weeks but diminished in week 28.

For the combined residues, the effect of gypsum dissolution was again apparent for the entire testwork period. In the first week, dissolution of natrojarosite to form ferric hydroxide appeared to be occurring as shown by the SI values. Week 28 showed the same effect but in reality iron was not detected so the SI's are maximum values and do not confirm that ferric hydroxide formed. Tenorite was over-saturated in the first week but not subsequently. Dissolution of carbonates also appeared to be an important effect.

In summary, the evaluation of saturation indices indicates that leachates were consistently in equilibrium with silica and gypsum and in the early stages of testing were probably also in equilibrium with iron and copper oxides. The interpretation did not provide any indication of possible constraints on the solubility of other potential contaminants such as cobalt, nickel and zinc. As a result, metal concentrations were compared to pH for all tests to evaluate pH control on metal concentrations.

Table 6-1: Saturation Indices for Humidity Cell Leachates

Mineral	Initial Release (week 1)								Release at Week 28							
	Leach Residue, no CuSO ₄	Leach Residue	Gypsum	Raffinate Neutralization Residue	Fe /Al	Mg Residue	Combined Residue	Comb. Residue, no Gypsum	Leach Residue, no CuSO ₄	Leach Residue	Gypsum	Raffinate Neutralization Residue	Fe /Al	Mg Residue	Combined Residue	Comb. Residue, no Gypsum
Hematite	2.7	2.7	5.2	6.9	10.0	13.4	13.6	13.5	3.3	2.4	5.9	7.2	8.2	10.4	10.4	10.4
Quartz	0.3	0.8	0.5	0.8	0.8	-0.7	0.8	-0.1	1.0	0.8	-0.3	-0.1	0.8	-1.5	1.2	1.1
Fluorite	-6.3	-3.2	-4.4	0.6	0.6	-0.2	-0.2	-0.5	-6.3	-6.6	-1.6	-1.5	-1.7	-0.9	-1.4	-1.4
Chalcedony	0.0	0.5	0.2	0.6	0.5	-0.9	0.5	-0.3	0.8	0.5	-0.5	-0.4	0.5	-1.8	0.9	0.9
Barite	-0.5	-0.7	-0.8	0.2	-0.5	0.0	-0.6	-0.3	-1.0	-1.2	-0.3	-0.1	-0.9	-0.8	-0.9	-1.1
Jarosite-K	-1.0	-0.9	-0.4	0.1	2.5	-5.4	-3.6	-4.3	-7.4	-6.6	-6.1	-4.7	-4.4	-7.9	-7.7	-7.6
Gypsum	-0.4	-0.3	-0.2	-0.2	-0.1	-0.1	-0.2	-0.2	-0.2	-0.2	-0.1	-0.2	-0.1	-0.1	-0.2	-0.1
Anhydrite	-0.6	-0.4	-0.4	-0.4	-0.3	-0.3	-0.3	-0.3	-0.3	-0.4	-0.3	-0.3	-0.3	-0.3	-0.3	-0.3
Amorphous Silica	-1.0	-0.5	-0.8	-0.4	-0.5	-1.9	-0.5	-1.3	-0.2	-0.5	-1.5	-1.4	-0.5	-2.8	-0.1	-0.1
Bassanite	-1.2	-1.1	-1.0	-1.0	-0.9	-0.9	-1.0	-1.0	-1.0	-1.0	-0.9	-1.0	-1.0	-0.9	-1.0	-0.9
Fe(OH)3(ppd)	-3.5	-3.5	-2.3	-1.4	0.1	1.8	1.9	1.9	-3.2	-3.7	-1.9	-1.3	-0.8	0.3	0.3	0.3
Jarosite-Na	-1.9	-2.5	-2.4	-2.0	0.8	-7.2	-5.2	-5.9	-9.4	-8.9	-8.8	-7.1	-8.3	-10.2	-9.8	-9.8
Epsomite	-2.2	-1.9	-3.5	-3.3	-1.9	-2.1	-2.1	-1.9	-5.4	-5.4	-6.6	-6.6	-6.4	-2.1	-4.1	-3.7
Tenorite	-5.7	-6.1	-5.0	-4.4	-1.2	1.1	0.7	0.9	-5.6	-6.7	-5.0	-5.4	-1.0	-1.3	-1.4	-2.0
Gibbsite	-6.4	-6.9	-4.7	-6.0	-3.3	-1.2	-0.5	-0.8	-1.5	-3.0	-3.2	-2.8	0.2	-1.0	-0.6	-0.7
Halite	-4.8	-3.9	-6.7	-6.6	-4.5	-4.3	-5.4	-4.4	-9.8	-9.5	-11.0	-10.5	-11.2	-9.7	-10.2	-10.0
Ni(OH)2(s)	-11.3	-11.7	-10.5	-8.5	-5.4	-1.6	-2.3	-2.5	-11.1	-12.3	-10.6	-10.2	-6.8	-5.4	-6.1	-5.7
Kaolinite	-11.8	-11.8	-7.9	-9.7	-4.6	-3.2	1.0	-1.3	-0.5	-3.9	-6.5	-5.3	2.4	-4.5	1.6	1.4
Brucite	-14.9	-13.7	-14.1	-12.3	-8.9	-0.3	-1.6	-0.8	-13.9	-14.9	-13.8	-13.3	-11.8	-3.2	-5.5	-5.0
Alunite	-10.2	-11.4	-8.0	-13.9	-8.2	-14.8	-11.4	-12.9	-2.6	-4.8	-10.3	-9.5	-1.8	-12.2	-10.7	-11.0
Cuprite	-20.9	-21.7	-19.2	-17.6	-12.3	-14.2	-14.9	-14.3	-15.7	-19.0	-12.0	-17.9	-11.1	-9.9	-13.1	-15.6
Talc	-31.9	-26.4	-28.5	-21.8	-11.9	8.3	10.3	9.1	-25.8	-30.0	-30.7	-28.4	-20.6	-4.1	-0.2	1.3
Dolomite ²	-	-	-	-	-8.8	3.5	2.5	2.9	-	-	-16.3	-	-12.4	-0.2	-1.5	-1.4
Dolomite-ord ²	-	-	-	-	-8.8	3.5	2.5	2.9	-	-	-16.3	-	-12.4	-0.2	-1.5	-1.4
Dolomite-dis ²	-	-	-	-	-10.3	1.9	0.9	1.4	-	-	-17.9	-	-14.0	-1.8	-3.1	-2.9
Calcite ²	-	-	-	-	-5.3	0.9	0.4	0.5	-	-	-6.7	-	-4.8	-0.9	-0.6	-0.7

Notes:

1. Green shading indicates saturation indices between -0.5 and 0.5. Amber shading indicates saturation indices greater than 0.5.
2. Carbonate not reported in leachates at low pH. Saturation indices for carbonate minerals not calculated.

6.1.2 pH Control on Metal Concentrations

The overall assumption in evaluating these plots is that if similar concentrations are indicated by tests operating at different liquid to solid ratios, and there is a relationship with pH that is consistent with first principles, it can be concluded that a solubility control is operating. If tests operating at low liquid to solid ratios yield higher concentrations than tests operating at high ratios, and the difference in concentrations can be accounted for by the ratio, it is more likely that the availability of soluble components is limited. The following bullets indicate observations for individual parameters.

Graphs are provided in Appendix D.

- Aluminum. A strong relationship between pH and Al concentration was defined mainly by humidity cell leachates but also by some MDNR reactor and shake flask leachates for the low pH samples. A strong control at neutral to basic pH was indicated by all test types.
- Arsenic. The leachate data indicated a pH relationship in which there was a pH minimum at about 6. Concentrations were comparable at both higher and lower pHs. At low pH, the relationship was defined by humidity cells. Lower concentrations were indicated for shake flask and MDNR reactor leachates. It is likely that solubility at acidic pHs is greater than indicated by the testwork. At pH greater than 6, arsenic concentrations were positively correlated to pH and the relationship was defined by all test types. The data are a good indication of arsenic constraints under these conditions.
- Cadmium. The results showed that cadmium was much more soluble under acidic conditions than neutral to basic conditions. The Fe/Al residues showed greatest solubility of cadmium at lowest pHs. Cadmium is probably more soluble at acidic pH than indicated by testwork. The presence of non-detectable cadmium at neutral to basic pH indicates that the detection limit is a reasonable indication of cadmium concentrations under these conditions.
- Cobalt. A strong relationship was shown for pH and cobalt concentrations. The bulk of the data describe increasing cobalt concentrations spanning several orders of magnitude as pH decreases from 9 to 3. The Fe/Al residues show a distinctive group at higher concentrations than the other residues. This group was defined by all three types of tests and implies a common solubility control for cobalt which is probably co-precipitated cobalt associated with iron oxides in this residue. Because this residue is also present in the combined residues and concentrations at neutral to basic pH are similar for all three tests, the data are a reliable indication of cobalt concentrations at neutral and moderately acidic pH when the Fe/Al residue is being leached.
- Chromium. Chromium results defined a strong pH relationship with all types of tests. Chromium concentrations showed a minimum at about pH 6 and higher concentrations for lower and higher pHs.

- **Copper.** The relationship for copper was similar to cobalt. At low pH, the Fe/Al residues showed the greatest concentrations in all test types. At higher pHs, copper concentrations were consistent for different residues. It appears likely that a copper oxide, co-precipitated with the iron oxide solids in the Fe/Al residue will control copper concentrations.
- **Iron.** Iron concentrations were mostly negatively correlated with pH except at the highest pH for which iron concentrations increased. Iron concentrations are higher than the ideal solubility of ferric hydroxide for pH greater than 4. This implies that ferric hydroxide was forming but that it formed as colloids which passed through the filter.
- **Manganese.** A strong negative correlation was apparent. Like Co and Cu, the Fe/Al residues showed the highest concentrations at lower pH implying that these residues were a source of co-precipitate. Because the relationship is indicated by all test types, the data appears to be a reliable indicator of manganese concentrations.
- **Molybdenum.** A positive relationship between molybdenum and pH was indicated for combined residues, Fe/Al residues and gypsum residues. The relationship at higher pHs was indicated by MDNR reactors and humidity cell samples.
- **Nickel.** The relationship for nickel was very similar to copper. The Fe/Al residues showed higher leachable nickel than other test types at acidic pH indicating that these residues with their elevated nickel concentrations probably contained co-precipitated nickel. Some leachates from the magnesium residues appeared to contain higher nickel concentrations than the combined residues possibly indicating a different solubility control (such as nickel hydroxide). The data for the combined residues which reflects the effect of leaching of Fe/Al residues imply lower nickel leaching from this control.
- **Lead.** Leaching of lead was apparent under acidic conditions but not neutral to basic conditions. Highest lead concentrations were apparent for the raffinate neutralization residue. The source of lead is unknown though this material contained the second highest lead concentration of any residue. Similar elevated lead concentrations were indicated for shake flasks and MDNR reactors but not for humidity cells.
- **Selenium.** Data for selenium showed that selenium leaching was greatest at higher pH and that concentrations were correlated with pH. The neutral to basic relationship was defined by all test types whereas under acidic conditions, greatest concentrations were shown by humidity cells implying that selenium was more soluble under these conditions than shown by the testwork.
- **Thallium.** In general, the relationship resembled nickel. The Fe/Al residue appeared to be the main source of leachable thallium and the relationship between pH and thallium concentration at neutral to basic pH was indicated by humidity cell and MDNR reactor leachates.
- **Zinc.** Like many other elements, the relationship for zinc appeared to be controlled by leaching of the Fe/Al residue as a source of co-precipitated zinc. Zinc concentrations at neutral to basic pH were consistently indicated by several different types of testwork.

Evaluation of metal concentrations with respect to pH indicates that under pH neutral to basic conditions, the concentrations indicated by humidity cells in combined residues are a reliable indicator of expected near equilibrium leaching. Maximum concentrations indicated in humidity cell leachates on combined residues (with gypsum) at neutral pH are shown in Table 6-2.

Leaching under acidic conditions is well-defined for the Fe/Al residue because solubility appears to be limited by the dissolution of iron oxides containing co-precipitated metal oxides of elements such as cadmium, cobalt, copper, manganese, nickel and zinc. For the acidic leach residue, upper limit solubility was not defined by the testwork; however, separate disposal of this waste is not contemplated.

Table 6-2: Summary of Maximum Concentrations Observed in Humidity Cell Leachates

Parameter	Concentration (mg/L)	Test Type
pH Range	6.6 to 8.5	All
SO ₄	7347	Humidity Cell
Al	0.18	Shake flask
As ¹	0.004	Humidity Cell
Cd	0.0004	Humidity Cell
Co	0.005	Humidity Cell
Cr	0.05	Humidity Cell
Cu	0.015	Humidity Cell
Fe	0.4	Humidity Cell
Mn	0.0023	Humidity Cell
Mo	0.14	Humidity Cell
Ni	0.098	Humidity Cell
Pb	0.0005	Humidity Cell
Se	0.054	Humidity Cell
Tl	0.0002	Humidity Cell
Zn	0.01	Humidity Cell

Notes:

1. Arsenic concentrations increased to 0.0075 mg/L as the test proceeded. The arsenic concentrations shown are for initial leachates for which maxima occurred for other parameters.

6.2 Trend Evaluation

6.2.1 Trends in Leachate Chemistry Shown by Testwork

As discussed in Section 5.3, various trends in leach chemistry have been observed. These include:

- pH which has trended steadily upward for most acidic residues and steadily downward for most non-acidic residues.

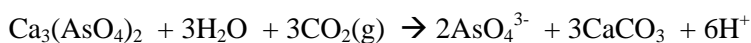
- Parameters showing general stable trends without apparent increases or decreases, for example sulfate and calcium for most test materials using all three test protocols. Some metals have also shown stable leaching.
- Parameters showing steady downward trends in concentrations, which include most metals.
- Parameters showing steady upward trends in concentrations, for example, arsenic in humidity cell leachates and boron in sequential shake flask and MDNR reactor tests.
- Undetectable trends due to concentrations below detection limits.

The main factors expected to contribute to trends in leachate chemistry are the presence of process solutions that presumably equilibrated with the solids prior to testing, the progressive dissolution of the solids by weekly additions of deionized water and removal of the leachate, and interaction between parameters that affect solubility (mainly pH).

The initial flushing of process waters typically yielded water containing high concentrations of sulphate, chloride, magnesium and sodium. Calcium concentrations were relatively low in the humidity cell leachates during this phase. Subsequent stable concentrations of some parameters can indicate that the solution is in chemical equilibrium with the solid(s) that are the source of the parameter, but also may indicate that the quantity available for dissolution during the leaching cycle is the same each week. The latter is not a chemical equilibrium. Sulfate is controlled by the dissolution of gypsum and SI's indicate that the solutions are in chemical equilibrium with gypsum in most cases. The exception is shown by the MDNR reactors and sequential shake flask tests on leach residues which yielded sulfate concentrations below the level expected for gypsum. This is probably a result of the lower concentrations of gypsum in this material.

General declining trends in metal concentrations may be due to shifts in pH (both downward and upward) and declining availability of readily leachable solids. Dissolution is controlled by available surface area which is expected to decline as the tests proceeded. In addition, as oxides age they become more crystalline trapping the co-precipitated metals and making the metals less leachable.

The upward trend in arsenic concentrations leaching from humidity cells containing combined residues occurs at low concentrations but appears to be related to the presence or absence of gypsum. The trend could be a result of breakdown of calcium arsenate which occurs as it equilibrates with carbon dioxide in the atmosphere:

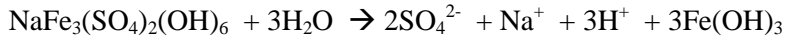


Calcium arsenate is not known to occur in the residues but arsenic concentrations are too low to allow detection of the compound directly.

6.2.2 Trends Beyond Testwork Time Frame

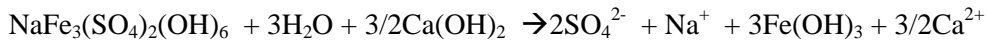
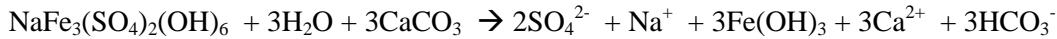
The proposed final disposal method will involve combination of all residues. The following discussion considers the long term pore water chemistry expected for the combined residues.

In the time frame of the tests, the combined residues were non-acidic because buffering capacity from brucite and possibly also calcite were present to offset the acidity produced by dissolution of natrojarosite:



Eventually, it is expected that acid buffering minerals will be exhausted and the residues will become acidic unless additional buffering capacity is added. This conclusion was confirmed by modeling the complete dissolution of all the mineralogical components of the residues in any of the proportions shown in Table 4-1 using React (Bethke 2005). As pH drops, metal mobility can also be expected to increase due to accelerated dissolution of the Fe/Al residue. Because the leach residue is dominantly natrojarosite and it is also the major component of the residue it is expected that acidification will result from dissolution of only a small proportion of the natrojarosite. However, under field conditions, dissolution of natrojarosite will be slow due to the slow movement of water through the residue mass.

PolyMet has proposed to add additional limestone or lime to the combined residues to ensure that they do not become acidic. The theoretical quantities of alkaline material needed can be calculated assuming the complete dissolution of natrojarosite balanced by calcium carbonate or hydroxide:



Using these reactions, the quantities of limestone or lime required are 6.2 and 2.3 g/kg per percent natrojarosite, respectively. This does not consider the effect of buffering by brucite from the Mg residue, which would reduce the requirement. These calculations assume the complete dissolution of natrojarosite. In reality, the process will not proceed to this endpoint because the conversion to ferric hydroxide will result in a decrease in volume and formation of iron hardpan. The latter will act as a barrier to water movement and dissolution of the residues.

6.3 Comparison of Results with Other Testwork Programs

As indicated in Section 2.2.1, other hydrometallurgical processes exist for recovery of metals from copper sulfide concentrates but none are directly comparable to the process proposed for the NorthMet Project. No comparisons with other testwork programs can be made.

6.4 Effect of Process Variations

The main process variation expected to occur is in the proportion of the various residues and the mineralogical composition of the residues. Changes in the proportion of leach and magnesium residues will result in a need to adjust the amount of basic material (i.e. limestone or lime) to be added. The magnesium residue would need to constitute about 50% of the combined residue to eliminate the need for additional basic material.

6.5 Conclusions

Characterization of hydrometallurgical residues has shown:

- Four of the residues (leach, gypsum, raffinate neutralization and Fe/Al) are expected to be acidic. The magnesium residue will be basic.
- The dominant mineral in the leach residue is jarosite, which generates acidic water when dissolved. Leach residue is the dominant component of the combined residues.
- The other residues (including magnesium) are mainly gypsum. The gypsum and raffinate neutralization residues are nearly entirely calcium sulfate. The Fe/Al residue also contains goethite, and the magnesium residue contains brucite.
- The Fe/Al residue also contains iron probably in amorphous form that contains co-precipitated metals.
- None of the residues were classified as hazardous wastes using the EPA 1311 protocol.
- Kinetic leach tests using three different protocols showed very similar results. Dissolution effects generally decreased with time though dissolution of gypsum exerted a very strong effect.
- Neutral to basic leachates produced by combined residues are believed to represent chemical saturation conditions suitable for prediction of pore water chemistry.
- The combined residue produced non-acidic leachate during the test but is expected to become acidic in the future unless additional basic material is added. PolyMet has proposed to add additional basic material as required to offset the acid potential of natrojarosite.

7 Pore Water Chemistry Prediction

7.1 Operational Model

7.1.1 Explanation of Modeling Approach

Because humidity cell test leachates had equilibrated with the residues, the testwork leachates are assumed to be an analogue for the residue pore water chemistry. No adjustment to the water chemistry is assumed to scale-up from test to site conditions.

7.1.2 Inputs to Water Quality Model

The main variable that could cause variations in water chemistry is inflow rate. However, because the concentrations in testwork leachates were found to be in equilibrium with the solids, concentrations will be independent of flow rate.

7.1.3 Results

Table 6-2 indicates predicted maximum expected concentrations in pore waters provided that pore waters remain non-acidic due to the addition of additional basic material to offset acidity produced by long term dissolution of natrojarosite. It is proposed that these are maximum values because they were produced by initial contact of leachates with the residues.

Leachate chemistry is expected to be dominated by the dissolution of gypsum, which will result in elevated sulfate concentrations. The presence of magnesium due to the dissolution of brucite ($\text{Mg}(\text{OH})_2$) will support higher sulfate concentrations than occurs for dissolution of pure gypsum.

The performance of combine residue without the gypsum residue is expected to be similar to combined residue with gypsum.

7.1.4 Conclusions

The chemistry of leachates in contact with combined residues in testwork has been estimated using results from leaching experiments.

7.2 Closure and Post-Closure Model

A separate prediction was not performed for closure conditions. Leachate chemistry as shown in Table 6-2 is expected to persist to closure. Slow movement of water through the residues is not expected to result in significant long term depletion of residues.

8 Conclusions

This report provides predicted pore water chemistry for the proposed disposal of combined residues in the hydrometallurgical residue cells. The results will be used to assess the requirement for treatment of residue leachates drained from cells at cell closure and the potential water quality impacts of leakage from the cells.

This report “**1UP005.01 – RS33/RS65 – Hydrometallurgical Residue Characterization and Water Quality Model – NorthMet Project - DRAFT**”, has been prepared by SRK Consulting (Canada) Inc.

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9 References

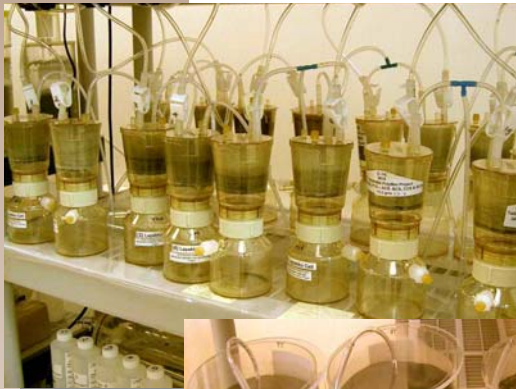
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Appendix A
Flotation Tailings and Hydrometallurgical Residue
Geochemical Characterization Plan



POLYMET
MINING CORP.

Flotation Tailings and Hydrometallurgical Residue Geochemical Characterization Plan NorthMet Project, Minnesota



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*Project Reference Number
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May 2006

Flotation Tailings and Hydrometallurgical Residue Geochemical Characterization Plan NorthMet Project, Minnesota

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1 Introduction

1.1 Background

PolyMet Mining Inc (PolyMet) is proposing to develop the NorthMet Project (Dunka Road Project of US Steel) near Babbitt, Minnesota. As a part of the Minnesota Department of Natural Resources (MDNR) "Permit to Mine" process a complete "mine waste characterization" will be required (Minnesota Rules Chapter 6132.1000). This document describes the plan developed for testing of flotation tailings and hydrometallurgical residue samples for the NorthMet Project.

The issues associated with tailings and residues at the NorthMet are expected to include acid rock drainage (ARD) and leaching of some heavy metals. The latter in particular are expected to include nickel and cobalt both of which do not require acidic conditions to be mobilized at elevated concentrations.

The objective of this program is to predict the reactivity of tailings and residues in their respective disposal areas for input into waste and water management planning, and environmental impact assessment. A separate Environmental Sampling and Analysis Plan describes characterization methods for effluents and emissions as metallurgical testing proceeds.

1.2 Geological Setting

The NorthMet Deposit is located in the intrusive mafic Duluth Complex of northern Minnesota. Disseminated copper-nickel-iron sulfides (chalcopyrite, cubanite, pentlandite and pyrrhotite) with associated platinum group element (PGE) mineralization will be extracted from several igneous stratigraphic horizons.

1.3 Agency Consultation and Design Process

This document was developed in consultation with staff from the Minnesota Department of Natural Resources (MDNR). The consultation included the following steps:

- June 22, 2005. A draft of the plan was prepared for MDNR Review.
- July 21, 2005. MDNR provided initial comments focussed primarily on flotation tailings.
- August 16, 2005. MDNR provided further comments on the characterization of hydrometallurgical wastes.
- September 9, 2005. SRK responded to the July 21, 2005 letter.
- September 14, 2005. A conference call was held to discuss the July 21, August 16 and September 9 letters.

This document includes responses to comments provided by MDNR, and has been prepared to conclude the design process and seek MDNR approval of PolyMet's plans to respond to the tailings/residue characterization component of requirements under Minnesota Rules 6132.1000.

1.4 Organization of This Document

This document describes:

- Section 2. Design basis for the program.
- Section 3. Analytical methods. This section describes methods used to analysis solids and leachates.
- Section 4. Use of the results in the context of water chemistry predictions.

1.5 Acknowledgements

The following individuals cooperated in the preparation of this plan:

- John Borovsky, Barr Engineering Company;
- Stephen Day, SRK Consulting;
- Paul Eger, MDNR;
- Jennifer Engstrom, MDNR;
- Don Hunter, PolyMet;
- Kim Lapakko, MDNR;
- Richard Patelke, PolyMet; and
- Jim Scott, PolyMet.

1.6 Laboratory Selection

The following laboratories will perform the procedures described in this plan (contact names for each laboratory are shown):

- ALS Chemex, North Vancouver, British Columbia – solids analysis listed in Section 4.1.1 (Bill Anslow);
- Optical – PolyMet or a Contractor (Richard Patelke);
- Sub-Optical Lab – McSwiggen and Associates (Peter McSwiggen);
- Canadian Environmental and Metallurgical Inc, North Vancouver, British Columbia – kinetic testing (Rik Vos); and
- Cantest Inc.. Vancouver, British Columbia - Kinetic test leachate analysis (Richard Jornitz).

2 Characterization Design

2.1 Objective of the Program

The overall objective of the program is to provide geochemical characterization information that can be used as inputs to design of management plans for the tailings and process residues and inputs into the environmental impact study (EIS) for the project.

2.2 Metallurgical Process Background

The processing and recovery of NorthMet Project ore to recover commodity metals will involve conventional flotation to produce a sulfide concentrate followed by hydrometallurgical treatment of the concentrate. The process flow sheets are provided in Appendix A.

Processing will result in the generation of the following waste products:

- Flotation tailings (low sulfide);
- Leach residue (silicates);
- Gypsum residue (calcium sulfate);
- Raffinate neutralization residue (calcium sulfate);
- Fe/Al residue (oxide); and
- Mg residue (oxide).

2.3 Tailings and Metallurgical Residue Disposal

Tailings and metallurgical residues are proposed for disposal in the existing impoundments at the former LTV Steel Mining Company (LTVSMC) operation (Figure 1). Review of the history of deposition at the LTVSMC tailings area indicates that the tailings are a result of processing ore feed from several iron ore pits and working faces in those pits. The ore was blended by truck delivery to loading pockets and then train delivery to crusher. Once in the plant ore was further blended in the coarse ore bins by a coarse ore tripper which continuously spread coarse ore across seven fine crushing lines and in the fine ore bins by a fine ore tripper which continuously spread fine ore across 34 mill lines. The fine ore was then processed through 34 mill lines in parallel. The tailings are a recombination from the 34 mill lines pumped to the basin and discharged at many spigots at the periphery of the basin. The tailings were deposited over many years as many layers in the basin.

Flotation tailings produced by conventional extraction of commodity-bearing sulfide minerals will be disposed in the existing Cells 1E, 2E and 2W. For the first five years of operation, cell 2W will be lined. Discharge methodology has not been determined but will most likely involve conventional discharge from one or more spigots. Hydrometallurgical residues produced by leaching of the sulfide concentrate will be disposed in lined basins within Cell 2W. The method used to transport the

residues to the cells has not been determined. The residues may be combined for disposal, or disposed separately depending on factors such as the possibility of selling some by-products. The possibility of constructing tailings dams using cycloned tailings is being considered and has been incorporated in this test program.

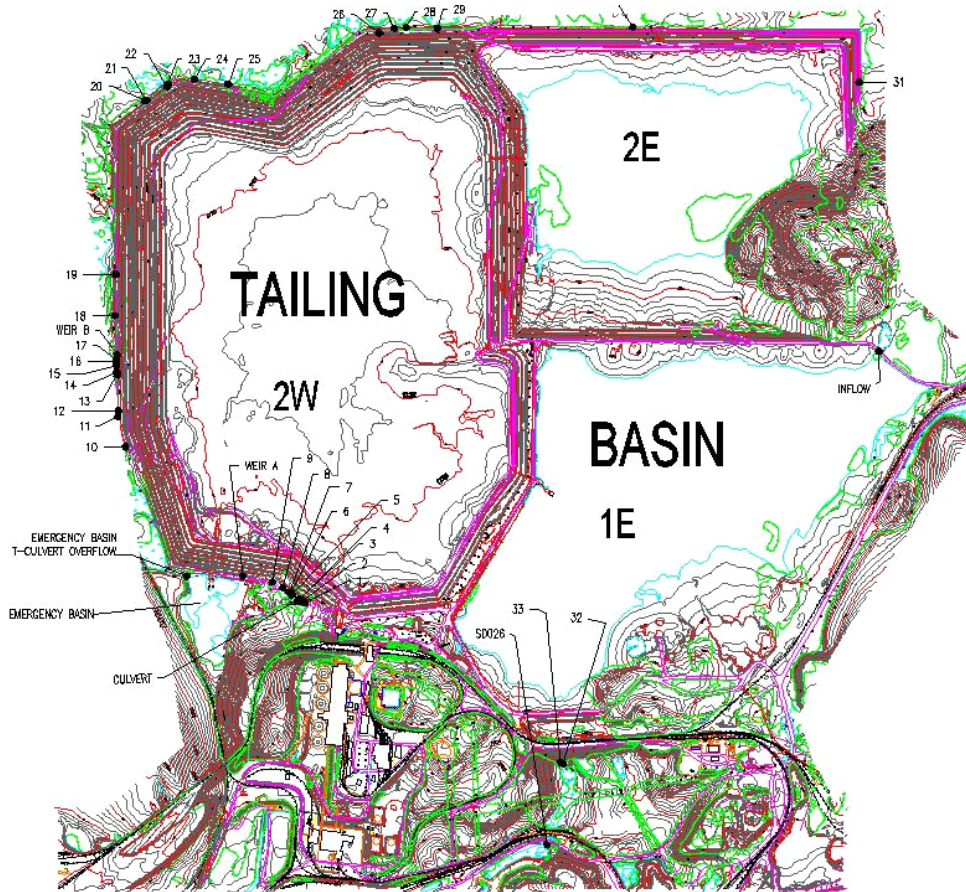


Figure 1: LTV Steel Mining Company Tailings Basins

2.4 Metallurgical Testing

Three ore composites were prepared under PolyMet’s direction from diamond drill hole core bracketing head grades expected during mining. Pilot-scale metallurgical testwork was begun in July 2005 and continued into September. Flotation tailings testing was completed in August and included assessment of process alternatives. Addition of copper sulfate to improve sulfide concentrate recovery was evaluated for two ore composites.

A separate Environmental Sampling and Analysis Plan has been prepared to describe monitoring of air, water and solid emissions as the testwork proceeds. That document explains the rationale for preparation of the three ore composites representing copper grades of 0.3%, 0.35% and 0.4%. It also contains details of pilot plant monitoring designed to evaluate variations in tailings geochemical characteristics potentially produced by variations in ore characteristics and process performance. For example, the pilot testing program included frequent (every two hours) monitoring of tailings characteristic.

2.5 Design Basis

2.5.1 Flotation Tailings

Flotation tailings will consist primarily of silicate minerals with small amounts of residual sulfide minerals not recovered by flotation.

The number of variables expected to control reactivity is relatively small. Ore processing naturally results in a well-blended tailings product because the process requires a uniform feed to maximize recovery of commodities. Therefore, variables such as rock type, sulfide mineral type, silicate mineral type and source of ore within the layers of the Duluth Complex will not be significant. Preparation of the ore composite, which has been documented by PolyMet mimics mining at several faces and will result in composites each containing similar distributions of the main silicate and sulfide minerals. Variations will occur reflecting the distribution of commodity-containing minerals. Further, the requirement for grinding to optimize beneficiation of the commodity minerals limits the importance of variables such as mineral particle size and degree of liberation. The remaining variables are therefore expected to be:

- Sulfur content;
- Metal content; and
- Particle size (where separations occur at the deposition site as a result of hydraulic factors) resulting in differences in chemical and mineralogical composition.

The testing of tailings from the three ore composites will allow these variables to be evaluated. It is expected that recovery of sulfide minerals from ore will vary during testwork resulting in tailings containing variable concentrations of sulfur and metals. Results of two-hourly testing were provided in a memorandum to DNR dated January 6, 2006 (Appendix B). The composition of the four tailings samples initiated concurrently with preparation of this plan are shown in Table 1 along with the range of sulfur concentrations indicated by the two-hourly testing.

Table 1: Composition of Four Tailings Samples

Ore Composite	Total Sulphur Content of Ore	Copper Sulfate Used in Flotation	Range of Total Sulphur Concentrations in Tailings	Total Sulphur Content of Tailings Under Test
1	0.86	NO	0.19% to 0.28%	0.23%
		YES	0.09% to 0.13%	0.10%
2	0.90	NO	0.05% to 0.25%	0.20%
3	0.86	YES	0.09% to 0.25%	0.15%

2.5.2 Hydrometallurgical Residues

The residues are mainly chemical products in which the original concentrate components are oxidized to sulfates and hydroxides, and dissolved.

- Sulfur from the sulfide minerals is oxidized to sulfate and precipitated as calcium sulfate through the neutralization process;
- Iron released from sulfide and silicate minerals is precipitated as hydroxide; and
- Magnesium and aluminum released from silicate minerals are precipitated as hydroxides.

Copper is recovered in the process by electro-winning to produce copper anodes. Nickel, cobalt and zinc are recovered as a mixed hydroxide product.

Incomplete dissolution of the silicate and sulfide minerals results in generation of a fourth mineralogical leach residue.

Since these products represent near-complete dissolution, oxidation and neutralization of the concentrate, the chemistry of contact waters is expected to be controlled mainly by the simple dissolution of the compounds. Oxidation of residual sulfide minerals may occur from the leach residue but this effect is expected to be minor.

Overall, the process of dissolution of neutralization products is unrelated to the composition of the ore and is not expected to vary in the long term except by depletion.

Pilot scale hydrometallurgical testing will be completed on two composite concentrate samples produced from processing of the three ore samples prepared with and without the use of copper sulfate in the flotation process.

3 Sample Handling and Analysis

3.1 Sample Shipping and Storage

SGS/Lakefield in Lakefield, Ontario, Canada is performing the metallurgical testing. Products will be shipped to Canadian Environmental and Metallurgical Inc. (Vancouver, British Columbia, Canada). Samples will be shipped and stored prior to testing as follows:

- Ore Feed Samples – Refrigerated;
- Sulfide Concentrate – Refrigerated;
- Flotation Tailings – Slurry in sealed pails with sufficient (6 cm) supernatant to ensure the samples are covered by water;
- Leach Residue – Cake, refrigerated; and
- Hydrometallurgical Residues – Cake, refrigerated.

Residual materials remaining after testing will be stored in the same condition as shipped.

3.2 Solids Characterization

3.2.1 Introduction

Table 1 summarizes the types of materials generated by testwork and the chemical testing procedures for each one. Physical testing of these products (including particle size determinations) is described in Environmental Sampling and Analysis Plan for the pilot plant test program. The DNR made several requests for multiple tests on some material types. In reality, the quantity of materials generated by metallurgical testing was limited and restricted the number of tests that could be performed. The 2-hourly testing showed that the sulfur content of the tailings did not vary widely and that the bulk samples under test will characterize the range of sulfur content of tailings.

Details of the test procedures are provided in the following sections.

Table 2: Procedures and Numbers of Samples for Testing

Material	Sulfur Forms	Neutralization Potential	Carbonate	Metals	TCLP	SPLP	Shake Flask	Optical Mineralogy	XRD	Sub-Optical	HCT	Sequential Shake Flask	Column	DNR Reactor Test	Layered Column
Ore feed	1/Ore		1/Ore	1/Ore	1/Ore	1/Ore		1/Ore	1/Ore	1/Ore	1/Ore				
Sulfide Concentrate	1/Ore		1/Ore	1/Ore	1/Ore	1/Ore		1/Ore	1/Ore						
Bulk Flotation Tailings – Without CuSO ₄ ¹	1/Ore		1/Ore	1/Ore	1/Ore	1/Ore		1/Ore	1/Ore	1/Ore	1/Ore			1/Ore	
Bulk Flotation Tailings – With CuSO ₄ ¹	1/Ore		1/Ore	1/Ore	1/Ore	1/Ore		1/Ore	1/Ore	1/Ore	1/Ore			1/ore	Multiple
Cyclone Sands	1/Ore		1/Ore	1/Ore	1/Ore	1/Ore		1/Ore	1/Ore	1/Ore	1/Ore			1/ore	
Tailings Slimes	1/Ore		1/Ore	1/Ore	1/Ore	1/Ore		1/Ore	1/Ore	1/Ore	1/Ore		2	1/ore	
Tailings Beaches	1/Ore		1/Ore	1/Ore	1/Ore	1/Ore		1/Ore	1/Ore	1/Ore	1/Ore		2	1/ore	
Leach Residue	1	1	1	1	1	1	1	1	1	1	1	1		1	
Gypsum Residue	1	1	1	1	1	1	1		1	1	1	1		1	
Raffinate Neutralization Residue	1	1	1	1	1	1	1		1	1	1	1		1	
Fe/Al Residue	1	1	1	1	1	1	1		1	1	1	1		1	
Mg Residue	1	1	1	1	1	1	1		1	1	1	1		1	
Reactive Residues ²	1	1	1	1	1	1	1		1	1	1	1		1	
Reactive Residue without Gypsum ³	1	1	1	1	1	1	1				1	1		1	

- Notes:
1. Flotation tailings samples were produced with and without the use of copper sulfate in the process to enhance recovery of sulfide minerals to the sulfide concentrate.
 2. Combination of leach, gypsum, raffinate, Fe/Al and Mg residues.
 3. Combination of leach, raffinate, Fe/Al and Mg residues.

3.2.2 Bulk Chemical Characterization

A split of each sample will be submitted for an extensive suite of analyses, as follows:

- Sulfur forms (total S, S as sulfate).
- Paste pH.
- Neutralization potential and carbonate.
- 50 elements (mostly metals by ICP scan following aqua regia (nitric and hydrochloric acids) digestion.
- Whole rock oxides. This provides total concentrations of major elements.

Method detection limits are provided in Appendix C.

3.2.3 Regulatory Leachate Characterization

All samples will be submitted for regulatory leach tests (EPA 1311; EPA 1312) to provide data for waste classification purposes should this be needed. Testing of the ore samples will provide a baseline for comparison to effects from processing.

3.2.4 Shake Flask

A third leach leachate procedure was used as the first step of the sequential shake flask leach procedure described in Section 3.3.3 for hydrometallurgical residues. This procedure, developed by Price (1997) for the British Columbia Ministry of Energy Mines and Petroleum Resources, uses a lower leach ratio (1:3) to improve detection of low levels of metals. The lixiviant is deionized water which typically has a pH between 5 and 6. The leachate is not fixed as in the TCLP and SPLP methods. The extraction is performed by shaking in a glass or plastic container for 24 hours, after which the leachate is extracted and analyzed.

3.2.5 Mineralogical Characterization

Mineralogical characterization will include:

- Optical Analysis on feed, product and mineral wastes (Flotation Tailings, Leach Residue). Other residues will not be examined optically since they are precipitates;
- X-Ray Diffraction on all samples to determine crystalline compounds; and
- Sub-Optical Analysis on ore feed, mineral wastes, and if practical residues to determine the distribution of trace elements in individual minerals prior to and following processing.

3.3 Kinetic Test Methods

3.3.1 Humidity Cell

Humidity cell testing will be performed on ore feed (to characterize three types of ore stockpiles), flotation tailings and leach residues using ASTM Procedure D 5744 – 96 (Reapproved 2001). This procedure was selected for the following reasons:

- Similar procedures have been in use under different names since the late 1980s (e.g. MEND 1991). The results can therefore be evaluated in the context of more than a decade of experience using the procedure.
- It is a standard procedure approved by the ASTM and is therefore defensible as a method.

The ASTM procedure provides some options for varying the test procedure. Appendix D provides a detailed listing of the requirements of the ASTM procedure, options chosen and any variances from the ASTM procedure.

3.3.2 MDNR Reactor

To allow comparison with previous MDNR studies, bulk tailings samples will be tested using a procedure referred to as the “MDNR Reactor” experiment. An apparatus specifically designed by MDNR (Appendix E) contains 75 g of solids.

3.3.3 Sequential Shake Flask Test

All residues are being tested using a sequential leach procedure consisting of weekly repetition of the leach procedure developed by Price (1997) in which the solid to liquid ratio is 1:3 (Section 3.2.4). The procedure involves weekly leaching of roughly 300 g of solids in a plastic bottle. The leaching step consists of addition of deionized water and agitation for 24 hours. The leachate is then decanted for analysis. Between leach steps, the bottle remains open to the atmosphere.

This procedure was requested by the DNR to provide a more aggressive evaluation of hydrometallurgical residue dissolution than occurs in humidity cells.

3.3.4 Leach Columns

A procedure to evaluate the interaction between leachate from NorthMet tailings and LTV tailings was designed and presented to the DNR (Appendix F). The procedure provides for two subaerial columns to generate leachate from the NorthMet tailings.

3.3.5 Leachate Analysis

Leachates from kinetic tests will be analyzed for the parameters indicated in Table 2, which also shows reporting limits. These limits are higher than the detection limits for the analytical instruments. Reporting limits represent the level at which the analytical laboratory (CANTEST) is

confident that the concentrations are quantifiable to an acceptable level. The instrument is able to detect much lower levels but these concentrations carry a very high degree of uncertainty which includes “undetectable”.

Low level leachate analyses for dissolved elements as shown in Table 2 are performed every four weeks (weeks 0, 4, 8 etc). On the intervening even numbered weeks (2, 6, 10 etc.), an ICP scan is performed using a higher detection limit primarily to determine the trend in major ions. pH and conductivity are determined every week. Acidity, alkalinity, inorganic carbon, sulphate, fluoride and chloride are determined every other week.

It is expected that testing of flotation tailings will result in very dilute leachates containing low concentrations of the metals of interest. Back-calculation of metal concentrations from other testwork performed by DNR indicates that cobalt and nickel concentrations could be in the tens of nanograms per litre (ng/L) for nickel and near nanograms per litre for cobalt. Quantification of these low metal concentrations is needed to provide reasonably constrained estimates of metals concentrations in the tailings storage facility.

A number of different approaches are available to quantify low levels of nickel and cobalt:

- The routine leachate analysis will achieve a reporting level of 0.0001 mg/L (100 ng/L). Should concentrations be undetected, detection limits of 50 ng/L can be obtained with additional processing effort using the same routine method.
- Specialist methods can achieve lower detection limits. These are non-routine (for example, evaporation to increase concentrations) and will need to be developed as the need arises.
- Existing testwork demonstrates that good correlations exist between cobalt and nickel concentrations in leachates. Detectable nickel concentrations can be used to estimate cobalt concentrations if this relationship can be demonstrated.
- In the event of undetectable low levels, a scale-up methodology will be agreed upon with MDNR to translate non-detectable concentrations to tailings concentrations. Detection limit values will be used in modeling calculations.

Table 3: List of Parameters for Low Level Analysis of Humidity Cell Leachates

Parameter	Reporting Limit	Parameter	Reporting Limit
pH (standard units)	-	Acidity	1
Conductivity (µS/cm)	1	Alkalinity	1
Chloride	0.2	Sulfate	0.5
Fluoride	0.05	Total Inorganic Carbon	1
ORP (mV)	-		
Dissolved Elements (mg/L)			
Aluminum	0.001	Mercury	0.00005 ²
Antimony	0.0001	Molybdenum	0.00005
Arsenic	0.0001	Nickel	0.0001 (0.00005) ¹
Barium	0.0001	Potassium	0.02
Beryllium	0.0002	Selenium	0.0002
Bismuth	0.0002	Silicon	0.05
Boron	0.005	Silver	0.00005
Cadmium	0.00004	Sodium	0.01
Calcium	0.01	Strontium	0.0001
Chromium	0.0002	Tellurium	0.0002
Cobalt	0.0001 (0.00005) ¹	Thallium	0.00002
Copper	0.0001	Thorium	0.0001
Iron	0.01	Tin	0.0001
Lead	0.00005	Titanium	0.0002
Lithium	0.0002	Uranium	0.00005
Magnesium	0.005	Vanadium	0.0002
Manganese	0.00005	Zinc	0.001

Notes: 1. Low detection limits are available for cobalt and nickel as shown.
 2. Lower level mercury analyses will be performed on selected samples.

3.4 Analysis of Remaining Sample Following Dissolution Tests

Analyses of the remaining sample following dissolution tests will be considered depending on the results obtained from the tests. Generally, these analyses can be of value if the test has undergone a major chemical change during the procedure (e.g. change from alkaline to acidic leachate) or if calculations indicate that a large quantity of one or more minerals or elements has been depleted. There is little value in post-test analysis if the depletion quantity is less than the uncertainty that can be expected from sampling of the test residue analysis. Experience also indicates that mineralogical analyses are also of little value unless weathering processes have had a detectable effect on the sample.

Therefore, residue analyses will be performed if:

- A large drop in pH has occurred (for example, from above 7 to below 5).
- Depletion calculations indicate that more than 10% of an important component was removed during the procedure.

Residue analyses will consist of the same procedures performed prior to the test.

3.5 Quality Assurance/Quality Control

A Quality Assurance Project Plan (QAPP) is being prepared for this project. To summarize, QA/QC includes the following components:

- Roughly 10% of all solids analyses will be performed in duplicate as sample availability permits.
- Roughly 10% of all cell and reactor tests will be run as duplicates if sufficient test material is available.
- A blank cell and reactor containing no sample will be operated to check for contamination of leachates by construction materials.
- Individual leachate results will be reviewed.
- Ion balances on leachate results will be reviewed. In general, imbalances of $\pm 10\%$ are considered acceptable. Re-analysis if requested depending on the nature of the imbalance.
- Data trends in kinetic test leachates will be analysed to check for anomalies.

4 Use of Data for Water Quality Predictions

4.1 Introduction

The data obtained from these programs will be used to estimate water quality during operation and closure. The following sections describe the application of the data to making water chemistry predictions.

4.2 Operational Water Quality

4.2.1 Flotation Tailings

Operational water quality in flotation tailings impoundments tends to be dominated by process water since this is the largest volume of water moving into and out of the impoundment by discharge and reclaim. Processes resulting from oxidation are not usually significant because continual placement of fresh tailings covers up older tailings before extensive weathering is initiated. The tailings pond water and trapped pore waters therefore reflect re-circulating process water. Seepage from the impoundments typically has a process water signature modified by anoxic conditions in the saturated tailings and interaction with LTVSMC taconite tailings.

Seepage chemistry will also be influenced by dam construction, particularly whether drains will be needed for stability. If drains are needed, water will be drained horizontally away from the dams, rather than the normal vertical seepage thru the coarse fraction of tailings near the dam.

The method used to estimate tailings pond water during operations is typically a coupled water and load balance that evaluates the effect of build-up of solutes in the tailings pond water due to interactions (e.g. mineral dissolution), reagent addition in the process, unintended additions in the process (e.g. Mo from lubricants), dilution due to rain and snowfall, dilution by run-in, and solute load loss due to encapsulation. If kinetic testing shows a short term leaching effect from beaches and dam faces, these loads are included.

The effect of interaction of saturated tailings with LTVSMC tailings will be evaluated directly by column tests (Appendix F). These tests will indicate whether any significant losses or additions occur as process water moves through the taconite tailings.

4.2.2 Leach Residues

Rinsed leach residues are expected to be relatively soluble and water chemistry associated with the residues will be dominated by equilibration of rinse. Water quality may be affected by the method of disposal (pumped slurry vs truck hauled solids). Since the residues will be continually accumulated, the operational water chemistry will be a result of mixing of rinsate water with precipitation and run-in. The chemistry of contact water during operation will be estimated directly from dissolution test results. Pore water chemistry indicated by testwork will be evaluated using MINTEQA2 or similar thermodynamic equilibrium models.

The effect of small amounts of sulfide minerals in the leach residues will be evaluated.

4.3 Water Quality at Closure and Post Closure

4.3.1 Flotation Tailings

At closure, the main effect is removal of inflows of process water and on-set of oxidation of tailings resulting in metal and possibly acidity loadings. The water and load balance developed for operational conditions is typically modified to evaluate these effects.

The long term closure modeling will need to consider evolution of the tailings profile in response to oxidation. This type of modelling uses humidity cell weathering rates, tailings physical characteristics, and moisture profiles predicted by modelling (HELP and HYDRUS-2D) as inputs. The propagation of the oxidation front and acidity front through the tailings is predicted and used to model the movement of solutes. The migration of the fronts can be used to estimate changes in solute loading in the future due to arrival of chemical fronts at the base of tailings. This type of modeling can be coupled with groundwater models to predict the chemistry of groundwater leaving the site.

4.3.2 Leach Residues

Water associated with the leach residues is expected to evolve as rinsate is displaced by dilute water from rainfall and snowmelt. Humidity cells will show whether this results in changing water quality. Concentrations indicated from humidity cells may be suitable for direct prediction of water chemistry or may be adjusted using MINTEQA2 or similar thermodynamic equilibrium models.

5 References

MEND, 1991. Acid Rock Drainage Prediction Manual. Mine Environment Neutral Drainage Program. Report 1.16.1b.

Nevada Division of Environmental Protection, 1996. Meteoric Water Mobility Procedure (MWMP) Standardized Column Test Procedure. NDEP publication MWMP.ltr. May 3 1996. 6p.

Price, W. 1997. Draft Guidelines and Recommended Methods for the Prediction of Metal Leaching and Acid Rock Drainage at Minesites in British Columbia. Reclamation Section, British Columbia Ministry of Energy and Mines. April 1997.

Sobek A A, Schuller W A, Freeman J R, and Smith R M., 1978. Field and Laboratory Methods Applicable to Overburden and Minesoils. USEPA Report No. 600/2-78-054, 203 pp.

Appendix A
Process Flow Sheets

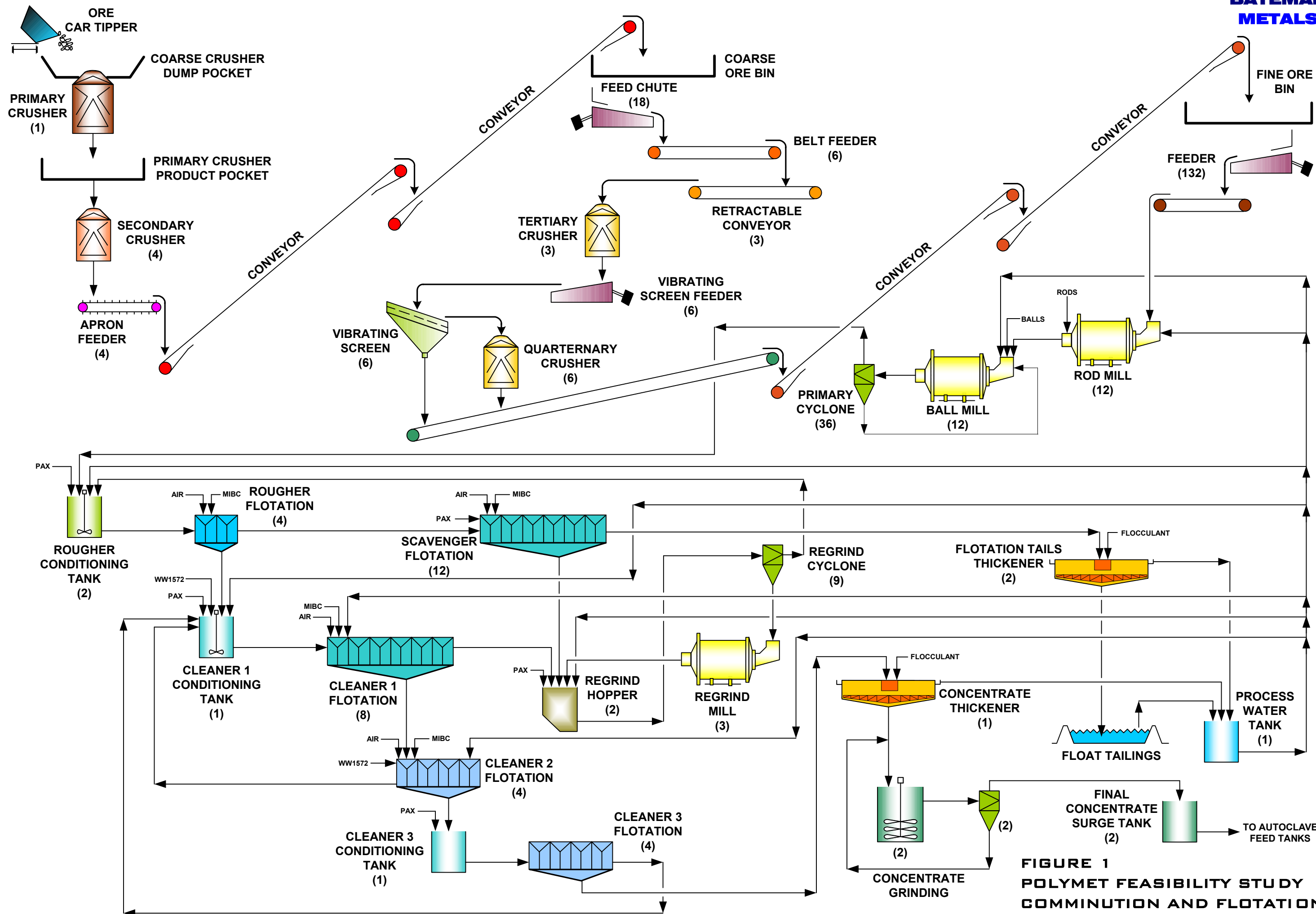


FIGURE 1
POLYMET FEASIBILITY STUDY
COMMINUTION AND FLOTATION
SCHEMATIC FLOW DIAGRAM

Appendix B
Update on Sample Selection from 24 Hour Testwork

Memo

To:	Jennifer Engstrom, MDNR	Date:	January 6, 2006
cc:	John Borovsky, Barr Jim Scott, PolyMet Don Hunter, PolyMet	From:	Stephen Day
Subject:	NorthMet Project Tailings and Hydromet Residue Testwork – Update on Sample Selection from 24 Hour Testwork	Project #:	1UP005.001

Jennifer

We have now received the 2-hourly tailings total sulfur analyses from the pilot plant testwork. These results allow final recommendations to be made for the selection of samples for tailings and hydrometallurgical testwork.

1 Results and Implications of 2-Hourly Sulfur Analyses

Results of the 2-hourly sulfur analyses are shown by the coloured solid lines in Figure 1. The broken horizontal lines are the concentration of sulfur in the composite tailings samples currently being tested in humidity cells. The sulfur content of the ore composites was very uniform (Parcel 1, 2 and 3, 0.86%, 0.9% and 0.86%, respectively) as shown by the solid black lines in Figure 1.

The trend in sulfur results in tailings is explained by the chronology of the testwork and evaluation of addition of copper sulfate as a reagent:

- Flotation testwork began on July 17 with Parcel 2 without the use of copper sulfate. Parcel 2 was processed entirely without using copper sulfate. As shown, sulfur concentrations varied from 0.05% to 0.25% reflecting adjustment of the process conditions early in the testwork. The average was 0.19%. The composite tailings sample has a sulfur content of 0.2% closely representing the average.
- Testwork continued with Parcel 1 without using copper sulphate. Processing was continuous so one point is shared between Parcel 2 and Parcel 1. The range of sulfur concentrations was 0.19% to 0.28% with an average of 0.24%. The composite sample was 0.23% and is close to the average.
- Pilot plant testwork was suspended on July 19 to allow for further bench scale testing on recovery of metals.
- The pilot plant resumed on August 8 using Parcel 1. Addition of copper sulfate was evaluated. This reagent causes activation of the sulfide mineral surfaces and improves bulk sulfide flotation. The effect of copper sulfate on tailings characteristics was immediately apparent for Parcel 1.

Total sulfur concentrations decreased to a range of 0.09% to 0.13% (average 0.1%) and the resulting tailings composite was 0.1%.

- Processing continued with Parcel 3 using the copper sulfate additive. Sulfur content of the tailings varied over a wider range (0.09% to 0.25%, average 0.18%) though the range was comparable to the total range indicated by processing of other ore packages. The resulting composite had a total sulfur content of 0.15%.

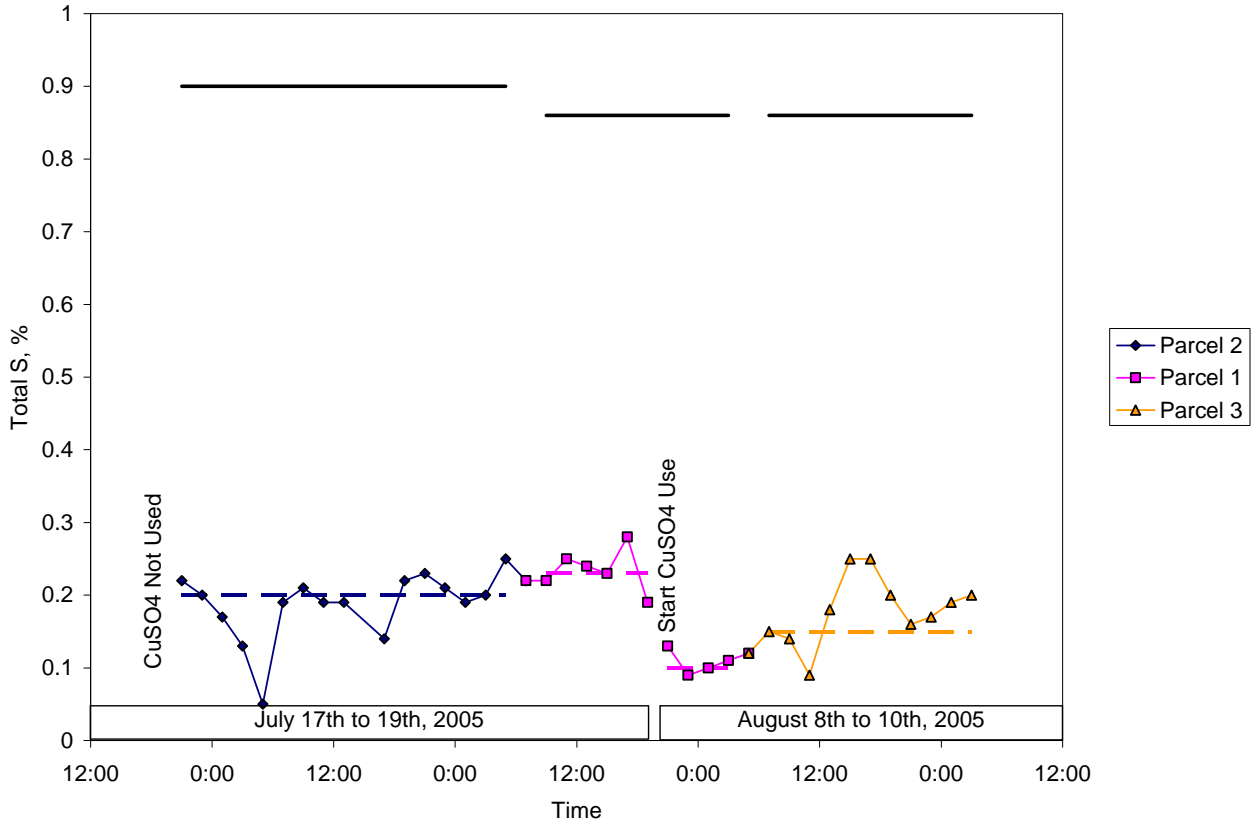


Figure 1. Results of 2-Hourly Total Sulfur Analyses. Solid lines and points connect 2-hourly results. Broken solid lines are sulfur concentrations in composite tailings samples representing each stage of testwork. Solid horizontal lines are the respective ore composite sulfur contents.

Based on the process testwork, Polymet has made the decision to advance the project with the use of copper sulfate to optimize overall sulfide mineral flotation. This decision is beneficial for the tailings since it is expected to lower the overall sulfide content.

The process testwork showed that sulfur concentrations in the tailings can be expected to vary in response to changes in process conditions including the use of copper sulfate. Parcel 3 showed that the use of copper sulfate may not always result in low sulfur content in tailings, and therefore there is a need to capture sulfur concentrations approaching 0.25% in the kinetic testwork. The samples generated without copper sulfate provide the required range and can be tested to represent the potential for higher sulfur concentrations in the tailings. The lack of copper sulfate for the Parcel 2 and 1 samples is not expected to have significantly affected the reactivity of the residual sulfide minerals in the tailings:

It is therefore concluded that:

- Kinetic testing of all four tailings samples should be continued.
- No additional samples are needed to represent the range of sulfur content expected in tailings.

2 Testing of HydroMet Residues

2.1 Source of Sulfide Concentrate for Hydromet Process Evaluation

Evaluation of the hydromet process was performed using two bulk sulfide concentrates produced by processing of ore parcels 2 and 1 (without copper sulfate) and ore parcels 1 and 3 (with copper sulfate). The sulfide concentrates contained the following total sulfur concentrations:

- No Copper Sulfate
 - Parcel 2 – 23.6%
 - Parcel 1 – 21.3%
- With Copper Sulfate
 - Parcel 1 – 22.1%
 - Parcel 3 – 21.6%

It is apparent that the sulfur content of the concentrates does not vary significantly though the effect of copper sulfate on concentrate sulfur content for Parcel 1 is apparent and corresponds with the matching decrease in sulfur content of the tailings. Since the decision has been made to proceed with the use of copper sulfate, only the residues produced from sulfide concentrate generated using copper sulfide should be tested.

2.2 HydroMet Residues

All the expected HydroMet Residues were produced by processing of the sulfide concentrate generated using copper sulfate.

A difference exists between the way that the residues were recovered in the pilot test compared to actual operating conditions.

To summarize, the first step in the process is the leaching of the sulfide concentrate to produce a low pH pregnant solution containing all the commodity metals. Subsequent recovery of the metals involves a series of pH adjustments to the leach solution that results in precipitation of products and residues. The products are then refined to recover the contained metals (copper, nickel, cobalt, PGM, zinc). The residues contain entrained leach solutions that have to be recovered to optimize recovery of commodity metals. Under full-scale operating conditions, recovery of the leach solutions from the residues will occur by rinsing the residue cakes with pH-adjusted re-cycled final process water to displace the leach solutions. The pH adjustment is required to ensure that metals in the leach solution are not lost to the solids.

However, under pilot plant conditions, the recycled process water was not available because the processing of the leach solutions occurred in a stepwise rather than continuous fashion. Each metal recovery step was performed and completed before proceeding to the next. The final process solution that will be used for rinsing at full-scale was only generated at the end of the pilot plant and was therefore not available for the residue rinsing steps. The difference between full-scale and pilot plant conditions represents a practicality of metallurgical testing in that operation the pilot plant continuously is not an option with the available quantity feed concentrate.

Residues generated by the pilot plant were rinsed with locally obtained river water. No additional rinsing of the residues is proposed for the dissolution testwork. Rinsing with river water was less aggressive in displacing metal-laden leach solutions than can be expected with pH-adjusted process water. The residues can therefore be expected to contain higher metal content than under operating conditions and indicate greater leachable metals in dissolution tests. The testwork will tend to over-estimate rather than under-estimate water quality for water management planning and impact assessment.

3 Conclusions

The following actions are proposed:

- Kinetic testing of all four existing tailings samples in dissolution tests will continue.
- Testing of residues produced by hydromet testing of sulfide concentrate (with copper sulfate) will be started as described in the “Flotation Tailings and Hydrometallurgical Residue Geochemical Characterization Plan”

Appendix C
Parameter Lists and Detection Limits for Analysis of Solids

ALS/CHEMEX METHOD CODE	ITEM	UINTS	CHEMEX DETECTION LIMIT
ME-ICP61 (four acid)	CU%	%	0.001
ME-ICP61 (four acid)	NI%	%	0.001
S-IR08 (LECO SULFUR)	S%TOT	%	0.01
ME-ICP61 (four acid)	S%ICP	%	0.01
PGM-ICP23 (30 GRAM)	PT_PPB	PPB	5
PGM-ICP23 (30 GRAM)	PD_PPB	PPB	1
PGM-ICP23 (30 GRAM)	AU_PPB	PPB	1
ME-ICP61 (four acid)	CO_PPM	PPM	1
ME-ICP61 (four acid)	AG_PPM	PPM	0.5
ME-ICP61 (four acid)	ZN_PPM	PPM	2
ME-ICP61 (four acid)	CD_PPM	PPM	0.5
ME-ICP61 (four acid)	MO_PPM	PPM	1
ME-ICP61 (four acid)	PB_PPM	PPM	2
ME-ICP61 (four acid)	AS_PPM	PPM	5
ME-ICP61 (four acid)	CR_PPM	PPM	1
ME-ICP61 (four acid)	V_PPM	PPM	1
ME-ICP61 (four acid)	TI%	%	0.01
ME-ICP61 (four acid)	AL%	%	0.01
ME-ICP61 (four acid)	CA%	%	0.01
ME-ICP61 (four acid)	FE%	%	0.01
ME-ICP61 (four acid)	K%	%	0.01
ME-ICP61 (four acid)	NA%	%	0.01
ME-ICP61 (four acid)	MG%	%	0.01
ME-ICP61 (four acid)	MN_PPM	PPM	5
ME-ICP61 (four acid)	P_PPM	PPM	10
ME-ICP61 (four acid)	BA_PPM	PPM	10
ME-ICP61 (four acid)	BE_PPM	PPM	0.5
ME-ICP61 (four acid)	BI_PPM	PPM	2
ME-ICP61 (four acid)	SB_PPM	PPM	5
ME-ICP61 (four acid)	SR_PPM	PPM	1
ME-ICP61 (four acid)	W_PPM	PPM	10

ALS/CHEMEX METHOD CODE	ITEM	UINTS	CHEMEX DETECTION LIMIT
ME-ICP41 (Aqua regia digestion)	CU%	%	0.001
ME-ICP41 (Aqua regia digestion)	NI%	%	0.001
ME-ICP41 (Aqua regia digestion)	S%ICP	%	0.01
ME-ICP41 (Aqua regia digestion)	CO_PPM	PPM	1
ME-ICP41 (Aqua regia digestion)	AG_PPM	PPM	0.2
ME-ICP41 (Aqua regia digestion)	ZN_PPM	PPM	2
ME-ICP41 (Aqua regia digestion)	CD_PPM	PPM	0.5
ME-ICP41 (Aqua regia digestion)	MO_PPM	PPM	1
ME-ICP41 (Aqua regia digestion)	PB_PPM	PPM	2
ME-ICP41 (Aqua regia digestion)	AS_PPM	PPM	2
ME-ICP41 (Aqua regia digestion)	CR_PPM	PPM	1
ME-ICP41 (Aqua regia digestion)	V_PPM	PPM	1
ME-ICP41 (Aqua regia digestion)	TI%	%	0.01
ME-ICP41 (Aqua regia digestion)	AL%	%	0.01
ME-ICP41 (Aqua regia digestion)	CA%	%	0.01
ME-ICP41 (Aqua regia digestion)	FE%	%	0.01
ME-ICP41 (Aqua regia digestion)	K%	%	0.01
ME-ICP41 (Aqua regia digestion)	NA%	%	0.01
ME-ICP41 (Aqua regia digestion)	MG%	%	0.01
ME-ICP41 (Aqua regia digestion)	MN_PPM	PPM	5
ME-ICP41 (Aqua regia digestion)	P_PPM	PPM	10
ME-ICP41 (Aqua regia digestion)	B_PPM	PPM	10
ME-ICP41 (Aqua regia digestion)	BA_PPM	PPM	10
ME-ICP41 (Aqua regia digestion)	BE_PPM	PPM	0.5
ME-ICP41 (Aqua regia digestion)	BI_PPM	PPM	2
ME-ICP41 (Aqua regia digestion)	GA_PPM	PPM	10
ME-ICP41 (Aqua regia digestion)	HG_PPM	PPM	1
ME-ICP41 (Aqua regia digestion)	LA_PPM	PPM	10
ME-ICP41 (Aqua regia digestion)	SB_PPM	PPM	2
ME-ICP41 (Aqua regia digestion)	SC_PPM	PPM	1
ME-ICP41 (Aqua regia digestion)	SR_PPM	PPM	1
ME-ICP41 (Aqua regia digestion)	W_PPM	PPM	10
ME-ICP41 (Aqua regia digestion)	TL_PPM	PPM	10
ME-ICP41 (Aqua regia digestion)	U_PPM	PPM	10

ALS/CHEMEX METHOD CODE	ITEM	UINITS	CHEMEX DETECTION LIMIT
ME-ICP06--whole rock geochemisrty by ICP-AES	SIO2	%	0.01
ME-ICP06--whole rock geochemisrty by ICP-AES	AL2O3	%	0.01
ME-ICP06--whole rock geochemisrty by ICP-AES	TIO2	%	0.01
ME-ICP06--whole rock geochemisrty by ICP-AES	FE2O3	%	0.01
ME-ICP06--whole rock geochemisrty by ICP-AES	CAO	%	0.01
ME-ICP06--whole rock geochemisrty by ICP-AES	MGO	%	0.01
ME-ICP06--whole rock geochemisrty by ICP-AES	MNO	%	0.01
ME-ICP06--whole rock geochemisrty by ICP-AES	NA2O	%	0.01
ME-ICP06--whole rock geochemisrty by ICP-AES	K2O	%	0.01
ME-ICP06--whole rock geochemisrty by ICP-AES	P2O5	%	0.01
ME-ICP06--whole rock geochemisrty by ICP-AES	BAO	%	0.01
ME-ICP06--whole rock geochemisrty by ICP-AES	SRO	%	0.01
ME-ICP06--whole rock geochemisrty by ICP-AES	LOI	%	0.01

Appendix D
Options and Variance in ASTM Humidity Cell Procedure

9. Sample Preparation

Section	ASTM Procedure Description	Description of CEMI Procedure NA – Not applicable to this Project ASTM – ASTM Procedure Followed	CEMI Variance from ASTM
9.1	Air dry as-received bulk samples of solid material to prevent the additional oxidation of reactive minerals or compounds. If air drying is not practicable, oven dry the solid material at a maximum temperature of $50 \pm 2^\circ\text{C}$ for 24 h, or until a constant weight is reached.	Samples were air-dried at room temperature ($\sim 20^\circ\text{C}$).	
9.1.1	If exploration-generated or run-of-mine solid material samples are not readily available, archived dried and crushed samples from geological exploratory or development drilling programs may be used for preliminary evaluations of ore and waste rock from new operations; this is provided that the available solid material samples are not significantly finer than 95 % passing a No. 12 (1.7-mm) sieve. Document the sample drying and preparation procedures used during the drill sampling program in order to interpret the results properly. Evaluate the effects of drying temperature on metals volatilization (for example, mercury in cinnabar vaporizes at temperatures exceeding 80 to 90°C) and mineral morphology and chemistry modifications (for example, on heating at temperatures exceeding 100°C , chalcocite changes crystal form and is oxidized subsequently from Cu_2S to CuO , CuSO_4 , and SO_2). Especially ensure that the effects of particle size distribution changes resulting from the more finely crushed sample are considered in the interpretation (this is, the potential for increased liberation of acid-producing and acid-consuming minerals with an attendant increase in mineral surface area).	NA	
9.1.2	In mining waste evaluations, the particle size for mill tailings will be significantly finer (commonly less than $150\ \mu\text{m}/100$ mesh) than the particle size distributions from ore and waste rock. Pilot plant tailings should be used if mill tailings are not available.	NA	
9.2	Screen the air-dried bulk samples through a 6.3-mm (1/4-in.) screen in accordance with Test Method E 276. Crush any oversize material so that 100 % passes the screen.	NA	

Section	ASTM Procedure Description	Description of CEMI Procedure NA – Not applicable to this Project ASTM – ASTM Procedure Followed	CEMI Variance from ASTM
Note 7	<p>Caution: Recent accelerated weathering studies of run-of mine waste rock from metal mines demonstrate that crushing a bulk sample so it passes a 6.3-mm (¼-in.) screen may change the character of the sample by artificially increasing liberation and consequent surface areas of acid-producing and acid-consuming minerals contained in the + 6.3-mm (¼-in.) material. A suggestion for avoiding this problem is to segregate the - 6.3-mm (¼-in.) fraction by screening rather than crushing, and to test that fraction according to the protocol and equipment described in this text. The + 6.3-mm (¼-in.) material can be tested separately (for example, Brodie, et al (10) describe a large-scale humidity cell test that would accommodate – 75-mm material). Samples from the drill core and cuttings also present material sizing problems, which must be considered when interpreting drill core and cuttings accelerated data. The drill core must be crushed to -6.3-mm (¼-in.) to fit the cell described in this test method. The resulting size distribution from crushing will differ from that of run-of-mine due to differences in fracture patterns inherent to blasting practices that produce run-of-mine material. By contrast, drill cuttings size fractions are commonly less than 6.3-mm (¼-in.) due to the rotary-percussive nature of obtaining the sample.</p>	NA	
9.3	<p>Mix and divide the bulk sample to obtain a representative test unit with a weight in the range of 8 to 10 kg, using a riffle splitter with 1-in. (2.54-cm) chutes. Divide the test unit into eight nominal 1-kg specimens. Seal each test specimen in a moisture-barrier bag.</p>	NA	
Note 8	<p>The dried sample should be mixed through the riffle splitter at least once before making any splits; recombine the splits resulting from the sample mixing exercise by pouring individual splits either over each other or through the splitter again. Once the actual split is made, it is wise to re-mix it (according to the above procedure) prior to making the next split.</p>		<p>Samples were mixed through the riffle splitter once.</p>
9.4	<p>Select one test specimen at random, and determine the moisture content by weighing and drying to constant weight at 80 ±5°C.</p>		<p>Determined at 20 °C</p>
9.4.1	<p>Crush the dried test specimen so that at least 95 % passes a 1.7-mm (10-mesh) screen, in accordance with Test Method E 276.</p>	NA	

Section	ASTM Procedure Description	Description of CEMI Procedure	CEMI Variance from ASTM
		NA – Not applicable to this Project ASTM – ASTM Procedure Followed	
9.4.2	Divide the crushed test specimen in half twice, using a riffle splitter with 6.35-mm (¼-in.) chutes, and select a ¼ subsample at random.	NA	
9.4.3	Transfer the selected subsample to a ring and puck grinding mill and grind to a nominal of 95 % passing a 150-µm (100-mesh) screen, in accordance with Test Method E 276. Use the subsample for chemical and mineralogical characterization of the test unit.	NA	
9.5	Select one test specimen at random, and determine the particle size distribution in accordance with Test Method E 276.	ASTM	
9.6	Select one test specimen at random for use in the accelerated test method. Divide the test specimen into four nominal 250-g subsamples using the riffle splitter with 25.4-mm (1-in.) chutes, and label and store in vapor-barrier bags until it is time to load the humidity cells.	NA	
9.7	Reserve the remaining test specimens for replicated testing or to resolve disputed results.	NA	

10. Apparatus Assembly

Section	ASTM Procedure Description	Description of CEMI Procedure NA – Not applicable to this Project ASTM – ASTM Procedure Followed	CEMI Variance from ASTM
10.1	<p>The humidity cells are table-mounted at a height sufficient to accommodate the placement of both the humidifier and one Erlenmeyer flask for effluent collection from the bottom of each cell. During the water-saturated and dry-air portions of each weekly cycle, feed air is metered to the bottom of each cell at the selected rate (1 to 10L/min). Feed air for the three-day dry-air portion is routed first through a desiccant column and then to each of the cells through a dry-air manifold. Feed air for the water-saturated air portion is routed through a water-filled humidifier by means of aeration stones or gas dispersion fritted cylinders/disks, and then to each humidity cell lid air exit port to prevent the short circuiting of air through cells containing more permeable solid material samples. A separatory funnel rack is mounted on the table that holds the cells if the weekly water leach is applied dropwise (drip trickle). Multiple separatory funnels (one for each cell) are held in the rack during the drip trickle leach that is performed on the seventh day of each weekly cycle. The separatory funnel can be used to meter the required water volume slowly down the sides of the cell wall until the sample is flooded if the weekly leach is to be a flooded leach.</p>	<p>Humidity cells are constructed of acrylic tubing with an inside diameter of four inches and an overall height of twelve inches, with an acrylic base plate. The base plate is glued to the tube and threaded with a nylon hose adapter to which a length of tubing is attached to allow for leachate drainage into a collection container. A perforated PVC support plate is positioned inside the cell, one inch above the base plate and covered with six layers of nylon mesh. A nylon adapter is threaded into the side of the cell between the support plate and the base plate and a length of tubing was connected from the side adapter to the humidifier to facilitate the inflow of humid air to the cell. A dry air line is also connected to each cell. Each cell is covered with a removable acrylic lid.</p>	<p>Approximately 16 cells per humidifier Flood leaching: peristaltic pump using a peristaltic pump Temperature: 20 ± 2°C. Feed air rate to be determined.</p>

11. Procedure

Section	ASTM Procedure Description	Description of CEMI Procedure NA – Not applicable to this Project ASTM – ASTM Procedure Followed	CEMI Variance from ASTM
11.1	Cell Loading:		
11.1.1	If more than one humidity cell is used at one time, label each with a sequential number, and use the same number for the matching collection vessel (Erlenmeyer flask).	ASTM	
11.1.2	Weigh each humidity cell (without its lid) and each collection vessel; record the tare weights of each to the nearest 0.1 g.	ASTM	
11.1.3	Cut the filter media (such as 12-oz/yd ² polypropylene described in 6.11) to the humidity cell's inside diameter dimensions so that it fits snugly yet lies flat on the perforated support.		Shark Skin filter paper (320mm)
11.1.4	Re-weigh the humidity cell, and record the resulting tare to the nearest 0.1 g; the original cell tare (11.1.2) minus the new cell tare is the weight of the filter media.	ASTM	
11.1.5	Transfer the contents from each of the four bags containing the 250-g samples (9.6) into the humidity cell. Prior to the transfer, mix the contents of each bag by gentle rolling to eliminate possible stratification that may have occurred during sample storage.	ASTM	
11.1.6	Re-weigh the loaded cell, and record the weight to the nearest 0.1 g; the loaded cell weight minus the combined cell and filter-media tare weight is the weight of the sample charge.	ASTM	
11.2	First Leach:		

Section	ASTM Procedure Description	Description of CEMI Procedure NA – Not applicable to this Project ASTM – ASTM Procedure Followed	CEMI Variance from ASTM
11.2.1	The first leach (whether drip trickle or flooded), designated as the Week 0 leach, initiates the 20-week long humidity cell test and establishes the starting or initial characteristics of the leachate. Either a 500-mL or 1-L volume of water may be used for the weekly leachates, depending on the weekly pore volume desired or the quantity of solution required for analytical purposes; however, once a weekly volume has been selected, that weekly volume must remain constant throughout the 20-week testing period. A centrifuged cell culture of <i>Thiobacillus ferrooxidans</i> may be used in the first leach in order to ensure that optimum conditions for accelerates weathering are present at the beginning of the test.	500 mL Flood Leach	
Note 9	In the testing of mining wastes, cation (including metals and trace metals) and anion loadings are commonly high in the Week 0 leachate due to the dissolution of pre-existing soluble oxidation salts present in the sample prior to sample collection. The average number of weekly accelerated weathering cycles required to flush these pre-existing salts ranges from 3 to 5 weeks. Oxidation products observed during these 3 to 5 weeks are principally from the pre-existing salts, while those products observed after this period are considered to be solely a function of the accelerated weathering procedure. A method for estimating the amount of pre-existing oxidation salts present in a solid material sample is described by Sobek, et al (6). A comparison of estimated salt storage data obtained using this method with the first three weeks of humidity cell effluent loadings from three different samples is describes by White and Jeffers (7).	NA	
11.2.2	Fill a separatory funnel with for each cell with de-ionized water using a volumetric flask. If the leach is to be performed using the drip trickle method, set each separatory funnel above its corresponding cell, and adjust the drip rate (approximately 3 to 4 L/min) so that the solid material sample is wetted thoroughly but not flooded.	NA	
11.2.3	A minimum of 2 to 3 h is commonly required to complete the drip trickle leach.	NA	

Section	ASTM Procedure Description	Description of CEMI Procedure NA – Not applicable to this Project ASTM – ASTM Procedure Followed	CEMI Variance from ASTM
11.2.4	If the leach is to be performed by flooding, the separatory funnel can be used to meter the selected water volume slowly down the sides of the cell wall until the sample is flooded. This application method reduces hydraulic agitation of the sample surface commonly caused by pouring liquid from an open-mouthed vessel. Alternatively, flooding may be accomplished by any application apparatus (for example, a peristaltic pump) that supplies the selected volume of leachant at a reasonable rate without causing agitation and suspension of the finer fractions contained in the sample charge.	ASTM	
11.2.4.1	Allow the flooded cell to sit for a period of 1 h before draining the leachate into the Erlenmeyer collection flask. The 1-h leach time commences after all of the leachant has been placed in the cell. The solid material sample should be saturated and covered with leachant to a depth sufficient to maintain sample saturation. In testing mining wastes, the observed depth of leachant cover from a 500-mL flooded leach performed in 10.2-cm (4.0-in.) ID cells is approximately 2.5 cm (1.0 in.).	ASTM	
11.2.5	The following is performed once the leaching process has been completed: to reduce the effects of evaporation, and to prevent the contamination of each cell by airborne contaminants, place the lids on their corresponding cells and let the cells complete the leachate draining process for the remainder of the leaching day and overnight.	ASTM	
11.2.6	Disconnect the cells on the day following the leach, and weigh and record the weight of each cell and Erlenmeyer collection flask. Set each filled collection flask aside for leachate analyses. (Measurements of pH and Eh and sample preservation procedures must be performed as soon as possible after leachate collection.) Return each cell, replace the filled collection flasks with clean, tared Erlenmeyer flasks, hook up all connections, and begin the dry-air cycle.	ASTM	
11.3	Dry-Air Cycle:		

Section	ASTM Procedure Description	Description of CEMI Procedure NA – Not applicable to this Project ASTM – ASTM Procedure Followed	CEMI Variance from ASTM
11.3.1	The commencement of the three-day dry-air period marks the beginning of each new weekly cycle of the accelerated weathering humidity cell test; the first full-week cycle after the first leaching is designated Week 1; subsequent weeks (commencing with the second dry-air period) are designated as Week 2, Week 3 ... Week n, etc.	ASTM	
11.3.2	To perform the dry-air cycle, feed air is metered to the humidity cell array with a flowmeter (see 6.3) set at a target rate in the range of 1 to 10 L/min per cell, depending on the objectives of the testing. The air flow rate must be checked daily and adjusted to the target value ± 0.5 L/min.	ASTM	
11.3.3	Feed air from the flowmeter is routed first through a desiccant column and then to each of the sells through a dry-air manifold. Air exiting the desiccant column should have a relative humidity of less than 10 % as measured with a hygrometer (see 6.23).	ASTM	
11.3.4	To maintain similar positive air pressure through the cells, attach a water-bubbling vessel to each humidity cell air exit port coming out of the humidity cell lid; a 50-mL Erlenmeyer flask with a rubber stopper containing a vent and air inlet tube serves as a simple and efficient bubbler.	ASTM	
11.3.5	The dry air is passed through each humidity cell for three days. Air flow rates from each of the cells should be checked each day, recorded, and adjusted, if necessary. See also Note 10.	ASTM	
11.4	Wet-Air Cycle:		
11.4.1	The three-day wet-air period commences on the fourth day of each weekly cycle.	ASTM	
11.4.2	To perform the wet-air cycle of the method, feed air is routed through a water-filled humidifier via aeration stones or gas dispersion fritted cylinders/disks and then to each humidity cell.	ASTM	
11.4.3	The water temperature in the humidifier is maintained at $30 \pm 2^\circ\text{C}$ to ensure that the sparged air maintains a relative humidity of approximately 95 % as measured with a hygrometer (see 6.23) from one of the humidifier exit lines. Air flow rates to each of the cells should be checked each day, recorded, and adjusted, if necessary.	ASTM	

Section	ASTM Procedure Description	Description of CEMI Procedure NA – Not applicable to this Project ASTM – ASTM Procedure Followed	CEMI Variance from ASTM
Note 10	It is good practice to measure the air flow rates and relative humidity of the air exiting each humidity cell during each day of the three-day dry- and wet-air periods; the measurements should be taken at the same time each day from the humidity cell air exit port; these measurements can be accomplished by installing a quick-disconnect fitting in the tubing that connects the air exit port to the bubbler.	NA	
Note 11	Coals spoils in eastern states are commonly saturated; Caruccio (10) has suggested the following geographic control alternative to the dry-air versus saturated-air scheduling: (1) Eastern States Samples – Six days of saturated air (versus three days dry/three days wet); and (2) Western States Samples – Three days dry/three days wet.	NA	
11.5	Subsequent Weekly Leaches:		
11.5.1	A second leach with water is performed on the day following the end of the three-day wet-air period (that is, day seven of the first weekly cycle). This leach marks the end of the first weekly cycle and is designated as the Week 1 leach.	ASTM	
11.5.2	Subsequent leaches are designated as Week2, Week 3 ... Week n, and they mark the end of the weekly cycle for that numbered week. Perform each weekly leach as described in 11.2.2 – 11.2.5. Weekly weighing of the test cells is optional.	ASTM	No weekly weighing of the cells.
11.6	It is recommended that the weekly accelerated weathering cycles described in 11.2, 11.3, 11.4 and 11.5 be performed for a minimum of 20 weeks.	ASTM	
Note 12	Additional weeks of accelerated weathering may be required to demonstrate the nature of the material, depending on the chemical composition of the solid material. For some metal mining wastes, researchers have shown that as much as 60 to 120 weeks of accelerated weathering data may be required to demonstrate the complete weathering characteristics of a particular sample (7, 12). The criteria for ending the testing may be site specific and should be agreed before initiating the testing.	ASTM	
11.7	Leachate Analyses:		

Section	ASTM Procedure Description	Description of CEMI Procedure NA – Not applicable to this Project ASTM – ASTM Procedure Followed	CEMI Variance from ASTM
11.7.1	Analyze the leachates for specific constituents or properties, or use them for biological testing procedures as desired, using (1) appropriate ASTM test methods or (2) methods accepted for the site where disposal will occur. Where no appropriate ASTM test method exists, other test methods may be used and recorded in the report, provided that they are sufficiently sensitive to assess potential water quality impacts at the proposed disposal site. Suggested minimum weekly analyses should include pH, Eh, conductivity, and selected metals could be analyzed less frequently (for example, at Weeks 0, 1, 2, 4, 8, 12, 16, and 20), especially if changes in leachate chemistry are slow. Whether visible phase separation during storage of the leachates occurs or not, appropriate mixing should be used to ensure the homogeneity of the leachates prior to their use in such analyses.	At the end of weekly cycle the volume of leachate collected is recorded. The leachate is filtered through a Gelman magnetic filter funnel fitted with a membrane filter with pore size of 0.45 microns and analyzed for the parameters listed in Table 2 of the RFP. Filtered leachate samples will be submitted to ALS Environmental/Cantest Ltd. for dissolved metals analysis as requested in Table 4 of the Waste Rock and Lean Ore Geochemical Characterization Plan. Conductivity, Eh, and pH are measured in the CEMI laboratory using standard procedures. An aliquot of filtered leachate is titrated with standardized sulphuric acid to pH 4.5 to calculate total alkalinity. Standardized sodium hydroxide is used to titrate an aliquot of leachate to pH 4.5 and to pH 8.3 to calculate total acidity. Analysis frequency: pH, cond, Eh every cycle; SO ₄ , Cl, F, alkalinity, TIC, acidity cycle 0, 2, 4, 6 etc.; ICP-MS including Hg and Si cycle 0, 4, 8, 12, etc., ICP-ES including Si cycle 2, 6, 10, 14, etc.	
11.7.2	Table 1 is an example of a spreadsheet format used for recording 20 weeks of leachate analytical data.	ASTM	
11.7.3	Fig. 5 is an example of a method used to plot the temporal variation (by week) of leachate pH, sulfate load, and cumulative sulfate load from 21 weeks of accelerated load and release rates).	ASTM	
11.8	Weathered Solid Material Analyses:		

Section	ASTM Procedure Description	Description of CEMI Procedure NA – Not applicable to this Project ASTM – ASTM Procedure Followed	CEMI Variance from ASTM
11.8.1	Weigh the humidity cell after collection of the final effluent and completion of a three-day dry-air period.	ASTM	
11.8.2	Transfer the weathered residue and filter media to a clean drying pan, and dry to constant weight at $50 \pm 5^\circ\text{C}$. Record the final weight.	ASTM	
Note 13	Perform any gross sample examination (for example, sample texture and weathering product mineralogic characterization) desired for the weathered residues prior to pulverization. To facilitate such an examination, empty the humidity cell contents into a clean drying pan carefully by pushing gently on the bottom of the perforated plate with a wooden dowel until the sample exits the cell mouth. The perforate plate is accessed through the humidity cell drain port.	NA	
11.8.3	Identify and mark the top versus bottom portions of the sample for gross sampling purposes. Formations of cemented lumps of sample termed “ferricrete” that result from the accelerated weathering process are common in iron-sulfide-mineral rich samples. Depending on the sample mineralogy, the degree of “ferricrete” cementation may vary vertically within the sample, and the investigator may wish to segregate the sample into upper, middle, and lower thirds to document and characterize such changes.	Procedure to be determined	
11.8.4	After drying to constant weight and prior to splitting, use an instrument such as a rolling pin to break up cemented lumps in the sample (if the cemented lumps cannot be sufficiently reduced to pass through the chutes of a riffle splitter, remove, record, and weigh separately):	ASTM	
11.8.4.1	Split the sample into halves using a riffle splitter with 2.54-cm (1-in.) chutes, and reserve one half to determine the particle size distribution in accordance with Test Method E 276.		Repeat same screen assay method as for pre-test characterization (s.9.5)
11.8.4.2	Split the remaining half sample into two quarters using a riffle splitter with 2.54-cm (1-in.) chutes, and submit one quarter for mineralogical characterization; pulverize the other quarter in either a ring-and-puck or disk-pulverizing machine to 95 % passing a 150- μm (100-mesh) screen in accordance with Test Method E 276.	Procedure to be determined	

Section	ASTM Procedure Description	Description of CEMI Procedure NA – Not applicable to this Project ASTM – ASTM Procedure Followed	CEMI Variance from ASTM
11.8.5	Mix the pulverized residue in a blender or on a rolling cloth. Use the prepared residue for chemical characterization and for comparison with the pre-weathered solid material sample.	Procedure to be determined	

Appendix E
Design of MDNR Reactor

Day, Stephen

From: Kim Lapakko [kim.lapakko@dnr.state.mn.us]
Sent: Tuesday, May 17, 2005 9:55 AM
To: Stephen Day
Cc: Dave Antonson; Jennifer Engstrom; Paul Eger
Subject: RE: Small reactor

Attachments: MN DNR psize methods 050517.doc



MN DNR psize
methods 050517.doc

Steve,

Attached is a description of the reactors, masses, and rinse volumes used for various size fractions of Duluth Complex rock in our particle size experiment. As indicated in the attachment, I won't have access to the trace metal data from that experiment until tomorrow. I will need to examine this to help evaluate the expected metal concentrations in drainage relative to detection limits. I'm not sure it will give us as much as hoped because the sulfur contents of the samples typically were on the order of 0.9% to 1.3%. This may make extrapolation by more than an order of magnitude tenuous. It will be another pertinent piece of information.

Kim

>>> "Stephen Day" <sday@srk.com> 5/17/2005 11:18:50 AM >>>
Dave

A design drawing should be fine along with description of the procedure.

The main question is what do you do to scale-up the sample mass as the particle size increases? I want to copy your procedure exactly.

Thanks
Steve.

-----Original Message-----

From: Kim Lapakko [mailto:kim.lapakko@dnr.state.mn.us]
Sent: Tuesday, May 17, 2005 8:38 AM
To: Stephen Day
Cc: Dave Antonson
Subject: Small reactor

Steve,

Dave Antonson will email a figure depicting our small reactor, along with some design details (perforated plate, adhesive, filter). He could also send a reactor. Please contact him directly, with an address to send it, if you think that would be helpful.

Kim

17 May 2005

Steve,

In our particle size tests we used a small reactor and 75-g mass for particle sizes of -270, +270/-100, and +100/-35 mesh. We used the ASTM cell and 1000-g mass for +35/-10, +10/-0.25 inch, and +0.25/-0.75 inch particle sizes. For rinse volumes, we used 200 mL for the 75-g samples and 300 mL for the 1000-g samples. The 300-mL rinse volume was determined as the quantity of water, rounded up to the nearest 100 mL, required to submerge the solids.

I won't have access to the metal release data for the particle size experiment until tomorrow. As mentioned on the phone, sulfate release rates appear to vary linearly with surface area. It seems likely that nickel release rates will vary similarly, and I'll look into this further tomorrow. Hopefully this information will shed some light on the maximum particle size question.

Day, Stephen

From: Dave Antonson [dave.antonson@dnr.state.mn.us]
Sent: Tuesday, May 17, 2005 11:53 AM
To: Kim Lapakko
Subject: reactor

Attachments: small reactor.doc

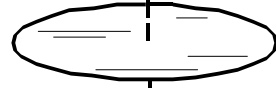


small reactor.doc
(271 KB)

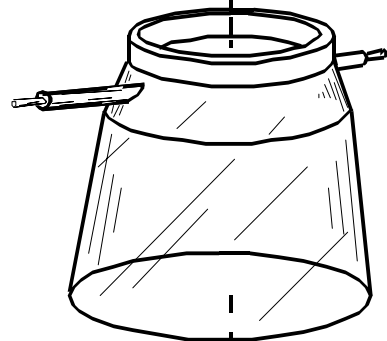
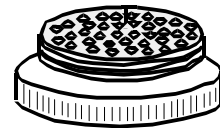
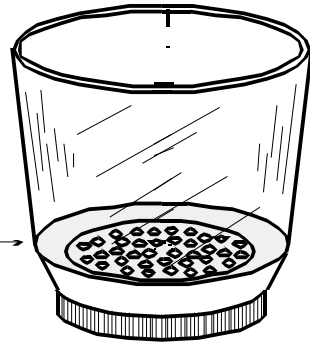
see if this makes any sense. you can edit it if you want. if it seems adequate you can forward it to steve. maybe he doesn't need a sample of the base.



1.6 micron glass
fiber filter



Perforated
plate



The reactors were purchased from Millipore Corporation (1-800-645-5476). They are 47 mm Sterifil aseptic systems. You will need the 250 ml receiver flask, 250 ml funnel (top), silicone o-rings, and the filter holder base and support screen.

The perforated acrylic plastic base was purchased as flat stock and fabricated to fit the top funnel. The plates are 1/8" thick, 2 1/4" in diameter and tapered to fit into the reactor top. Approximately sixteen 1/16" holes were drilled in the plate. The plate was glued into the reactor using acrylic solvent cement purchased from United States Plastics (1-800-537-9724). Catalog # 44629 for 5 oz. tube. The acrylic flat stock was also purchased from United States Plastics.

After the plate is glued into the top of the reactor there should be approximately a 3/8" gap between the bottom of the perforated plate and the top of the support screen of the filter unit.

The filter that rests on the perforated plate is a 55 mm Whatman GF/A glass microfibre filter (catalog # 1820 055).

Note: Before adding the solids to the filter you should wet the filter slightly with distilled water so no solids escape around the filter.

Appendix F
Design of Column Testing on Interaction between
NorthMet and LTV Tailings

Memo

To:	Jennifer Engstrom, MDNR	Date:	December 8, 2005
cc:	Kim Lapakko, MDNR Paul Eger, MDNR	From:	Stephen Day
Subject:	Design of Column Testing on Interaction Between NorthMet and LTV Tailings	Project #:	1UP005.001

Jennifer

During our conference call on Oct 25, 2005, I agreed to provide design details for column testing to evaluate the effect of contact of leach waters from the NorthMet Tailings with LTV tailings when we received characterization data for the samples obtained from the LTV impoundment in September. Complete results have now been received.

This memorandum describes:

- Characteristics of LTV tailings.
- Characteristics of available tailings sample material.
- Proposed testwork.

The original proposed program was described in a memorandum dated September 23, 2005. SRK is seeking comments from MDNR on this proposal. It is acknowledged, as we discussed during conference call that this column testwork may represent a preliminary assessment. Additional testing may be required as the design for the tailings basin progresses.

1 Characteristics of LTV Tailings

1.1 Sample Analysis

Seven holes were drilled in the tailings to a depth exceeding 60 feet using a geoprobe. Samples have been analyzed from five holes with tailings having the following textural characteristics:

- GP-1 – Mainly coarse sand.
- GP-2 – Interlayered fine sand and slimes.
- GP-3 – Coarse sand grading into fine sand and slimes.
- GP-4 – Interlayered coarse and fine sands.
- GP-5 – Interlayered fine sand and slimes.

Samples were obtained as core and shipped whole to Canadian Environmental and Metallurgical Inc. At the laboratory, all discrete textural layers were tested for rate of HCl reaction (ie a “fizz” test) and qualitative magnetism as an indicator of magnetite content. Samples were selected from each hole to represent the surface material (ie potentially weathered) and two samples of each textural type from each hole. These samples were submitted for relative density, moisture content and particle size determinations, quantitative mineralogy by x-ray diffraction and chemical analysis.

1.2 Results

Table 1 shows selected data sorted by the main textural groups (coarse sand, fine sand and slimes).

The dominant mineral in all samples was quartz which varied from 58 to 79% (by weight) but was not different in the three textural groups. Hematite and magnetite were present as expected. Magnetite was lower in the slimes samples likely resulting from density segregation as the tailings were deposited. Carbonates were a significant mineralogical component varying from 5 to 14%. Total carbonate content was greater in the slimes fraction compared to the coarse sands. Ankerite and siderite dominated and occurred in about equal amounts. The calcite content was lower than either ankerite or siderite.

Pyrite was detected in most samples but at very low levels. The sulphur content of the samples varied from 0.02 to 0.04% equivalent to pyrite content of 0.04 to 0.08%.

Silicates occurring in all samples were hydrobiotite, kaolinite, amphibole (cummingtonite ± grunerite), diopside, ferripyrophyllite (possibly minnesotaite) and albite. Other minerals occurring in a few samples were pyrophyllite, muscovite and hydroxylapatite. There was no evidence that the mineral distribution was related to particle size.

Distribution of metals was also unrelated to particle size with the possible exception of manganese which appeared to be elevated in the slimes. This is consistent with the higher carbonate content and indicates that manganese is associated with the carbonates.

2 Characteristics of PolyMet Tailings

Four tailings samples have been tested. These samples originated from processing of three ore composites nominally containing 0.35%, 0.4% and 0.45% copper (Parcels 1, 2 and 3, respectively). Flotation testing also considered addition of copper sulphate as an activator of pyrite. For Parcel 1 and 3, copper sulphate was evaluated. Also for Parcel 1, and Parcel 2 processing without the addition of copper sulphate was evaluated. The addition of copper sulphate does not materially affect the copper and sulphur content of the tailings directly but changes the mineralogical composition due to the selective recovery of pyrite to the bulk sulfide concentrate.

The characteristics of the four samples are shown in Table 2. Very little difference between the tailings was apparent. As expected, addition of copper sulphate in the process resulted in lower sulphur content in the tailings (0.1% and 0.15%, Parcels 1 and 3, respectively) compared to 0.23% and 0.2% (Parcels 1 and 2, respectively) without copper sulphate. Copper showed the widest variation in concentration (223 to 527 mg/kg). Variations of cobalt, nickel and zinc were small.

Table 1. Characteristics of LTV Tailings Samples

Core	Sampled		Material	Q %	Py %	Cal %	Ank %	Sid %	Hem %	Mag %	Bio %	Kao %	Fe- Pyr %	Albite low %	As ppm	Cd ppm	Co ppm	Cr ppm	Cu ppm	Fe %	Mn ppm	Ni ppm	P ppm	Pb ppm	S %	Zn ppm	
	Start ft	Finish ft																									
Coarse Sand																											
GP-1	8	12	Coarse Sand	72	0.2	0.3	3	3	3	4	1	1	2	2	31	-0.5	12	100	14	15.85	5970	5	240	7	0.02	14	
GP-1	20	40	Coarse Sand	71	0.2	0.2	5	8	2	2	2	1	2	2	36	-0.5	14	90	25	15.5	7110	8	250	5	0.04	13	
GP-3	8	12	Coarse Sand	58	0	0.4	6	6	2	3	5	1	6	4	16	-0.5	7	42	7	13.7	3420	3	250	4	0.02	9	
GP-4	4	16	Coarse Sand	59	0.1	0.7	2	4	2	3	10	2	2	3	19	-0.5	7	57	9	15.45	3890	1	240	7	0.02	9	
GP-4	20	24	Coarse Sand	79	0	0.1	3	2	2	3	3	1	2	1	21	-0.5	10	45	8	12.85	4010	3	250	5	0.02	34	
n				5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	
Min				58	0	0.1	2	2	2	2	1	1	2	1	16	-0.5	7	42	7	12.85	3420	1	240	4	0.02	9	
Median				71	0.1	0.3	3	4	2	3	3	1	2	2	21	-0.5	10	57	9	15.45	4010	3	250	5	0.02	13	
Max				79	0.2	0.7	6	8	3	4	10	2	6	4	36	-0.5	14	100	25	15.85	7110	8	250	7	0.04	34	
Fine Sand																											
GP-1	60	72	Fine Sand	72	0.1	0.7	3	2	1	1	2	2	2	4	22	-0.5	11	77	20	12.05	7010	3	330	3	0.04	14	
GP-2	0	1	Fine Sand	60	0	0.2	6	7	2	2	11	4	3	0	15	-0.5	9	27	7	14.4	4270	-1	490	-2	0.02	10	
GP-2	24	28	Fine Sand	62	0.4	1	6	3	1	2	5	2	3	3	22	-0.5	11	61	13	14.55	5340	3	550	7	0.02	14	
GP-3	44	60	Fine Sand	68	0.2	0.5	4	3	3	2	4	1	3	1	15	-0.5	12	45	17	13.4	8510	4	400	7	0.03	12	
GP-4	18	20	Fine Sand	73	0	0.2	7	3	2	2	4	2	2	1	43	-0.5	10	41	12	13.3	4020	2	290	8	0.02	13	
GP-5	8	20	Fine Sand	78	0.1	0.1	4	2	3	3	3	1	2	1	14	-0.5	7	42	7	13.45	3630	2	270	2	0.02	7	
GP-5	36	48	Fine Sand	73	0.4	0.4	4	2	2	3	2	1	3	2	14	-0.5	9	53	14	13.55	5820	2	290	4	0.02	8	
n				7	7	7	7	7	7	7	7	7	7	7	7	7	7	7	7	7	7	7	7	7	7	7	
Min				60	0	0.1	3	2	1	1	2	1	2	0	14	-0.5	7	27	7	12.05	3630	-1	270	-2	0.02	7	
Median				72	0.1	0.4	4	3	2	2	4	2	3	1	15	-0.5	10	45	13	13.45	5340	2	330	4	0.02	12	
Max				78	0.4	1	7	7	3	3	11	4	3	4	43	-0.5	12	77	20	14.55	8510	4	550	8	0.04	14	
Slimes																											
GP-2	28	32	Slimes	62	0	1	4	4	2	1	5	2	3	5	18	-0.5	14	31	10	14.2	7390	3	530	4	0.02	13	
GP-3	60	72	Slimes	62	0	0.7	8	5	3	1	7	2	2	1	15	-0.5	14	33	20	12	10050	4	590	2	0.04	14	
GP-5	20	24	Slimes	70	0.1	0.3	6	4	3	2	6	2	3	1	25	-0.5	9	38	7	13.75	4830	1	460	4	0.02	8	
GP-5	48	52	Slimes	72	0	0.6	4	6	2	1	4	1	2	1	16	-0.5	16	40	19	13.1	12400	3	550	4	0.03	11	
n				4	4	4	4	4	4	4	4	4	4	4	4	4	4	4	4	4	4	4	4	4	4	4	
Min				62	0	0.3	4	4	2	1	4	1	2	1	15	-0.5	9	31	7	12	4830	1	460	2	0.02	8	
Median				66	0	0.65	5	4.5	2.5	1	5.5	2	2.5	1	17	-0.5	14	35.5	14.5	13.425	8720	3	540	4	0.025	12	
Max				72	0.1	1	8	6	3	2	7	2	3	5	25	-0.5	16	40	20	14.2	12400	4	590	4	0.04	14	

Notes

Minerals Q = Quartz, Ank = Ankerite, Hem = Hematite, Mag = Magnetite, Sid = siderite, Bio = hydrobiotite, kao = kaolinite, Fe-Pyr = Ferriprophyllite, Cal = Calcite, py = pyrite

Table 2. Characteristics of NorthMet Tailings Samples

Sample Number	Source and Process	Total S %	As ppm	Cd ppm	Co ppm	Cr ppm	Cu ppm	Mn ppm	Ni ppm	P ppm	Zn ppm
PIS-ACS TO DSC COMP	Parcel 1, CuSO ₄ Added	0.10	<5	<0.5	59	186	223	1130	319	790	95
PISA TO D COMP	Parcel 1, CuSO ₄ Not Added	0.23	<5	<0.5	62	188	248	1140	329	800	97
P2SA TO D COMP	Parcel 2, CuSO ₄ Not Added	0.20	<5	<0.5	61	199	527	1125	385	750	93
P3S-A TO D COMP	Parcel 3, CuSO ₄ Added	0.15	<5	<0.5	60	175	418	1110	372	770	92

3 Proposed Column Testwork

3.1 Design Basis

Based on the characteristics of the NorthMet and LTV tailings, the following chemical processes can be expected to occur within the layered tailings basins:

- In NorthMet Tailings
 - Near surface oxidation of residual sulfide minerals resulting in release acidity, iron, sulphate and trace elements (copper and nickel).
 - Development and migration of an oxidation front due to consumption of oxygen near the surface.
 - Attenuation of metals as a result of interaction between pore fluids and mineral grains.
- LTV Tailings
 - Enhanced dissolution of ankerite and siderite under saturated conditions resulting in release of calcium, magnesium, ferrous iron, reduced manganese and bicarbonate alkalinity.
 - Localized re-precipitation of ferric hydroxides and manganese oxides due to variations in pH and oxidation-reduction potential.
- Interaction Between NorthMet Tailings Pore Water and LTV Tailings
 - Possible sorption of metals by ferric hydroxides and manganese oxides, particularly in the immediate contact zone where LTV tailing are probably partially oxidized.
 - Precipitation of metal carbonates due to alkaline conditions.

Testing of the LTV tailings indicates little significant variation in mineralogical and chemical content. The primary variable expected to influence the degree to metal attenuation occurs, if at all, is particle size which will control the availability of adsorption sites, oxidation-reduction conditions and contact time.

3.2 Proposed Testwork

The proposed leach column design includes the following main features (Figure 1):

- In series leach columns designed to generate NorthMet tailings pore water as feed into LTV tailings.

- First column containing 10 kg of unsaturated NorthMet Tailings and second receiving column containing 5 kg of LTV tailings. The larger volume of NorthMet tailings is intended to optimize development of near equilibrium pore water chemistry below an oxidation zone in the NorthMet tailings. The smaller volume of LTV tailings may allow breakthrough of NorthMet tailings water chemistry to be observed.
- NorthMet tailings open to atmosphere.
- Connecting pipe between columns and sampling “T” operated to exclude oxygen.
- Side sampling port in LTV tailings column to enable sampling of pore water just below the entry point.
- Sampling of final effluent.
- Application of 2 L of deionized water every week to allow withdrawal of up to 250 mL of water from each of the two intermediate location and 1.5 L of the final effluent. This application rate represents approximately one pore volume every 4 weeks.
- Analysis of intermediate sampling points for pH and Eh every week and composite sample for anions and cations every other week (including sulfur).
- Analysis of final effluent for same parameters, sulphate, alkalinity and anion scan.

Approximately 11 kg of each of the tailings samples shown in Table 2 are available for additional testing. The following matrix summarises six proposed tests (Table 3). It is preferred to test slimes and coarse sands as two extreme characteristics of LTV tailings. From experience, it is unlikely that the slimes will transmit sufficient water for the experiment. Therefore, a fine sand composite would be used instead.

Each “X” in Table 3 represents an in-series column pair. The two control experiments will operate without NorthMet tailings in the first column to evaluate leachate chemistry from LTV tailings. This will allow comparison with seepage chemistry in the existing basin.

Table 3. Matrix of Proposed Tests

		NorthMet Tailings Samples		
		Control No NorthMet Tailings	Lower S Composite (Parcels 1 and 3, CuSO ₄ Added)	Higher S Composite (Parcels 1 and 2, CuSO ₄ added)
LTV Samples	Fine sand or Slimes Composite	X	X	X
	Sand Composite	X	X	X

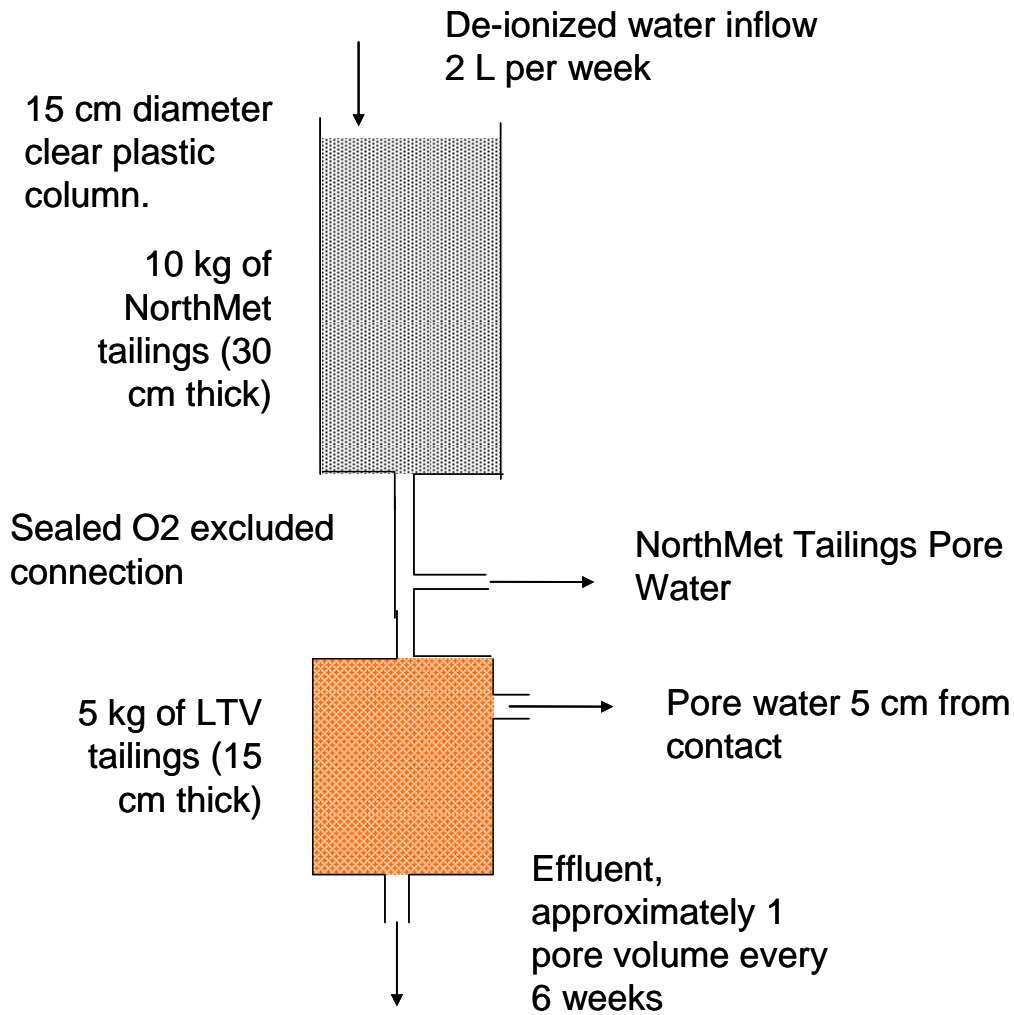


Figure 1. Schematic of In-Series Column Design

Appendix B
Leachate Extraction Test Results

Procedure	Parameter	Unit	Residues							
			Leach, no CuSO4	Leach, with CuSO4	Gypsum	Raffinate Neutralization	Fe/Al	Mg	Combined	Combined no Gypsum
SPLP Leachate Extraction Results	pH	-	2.79	3.38	4.12	4.91	5.03	9.72	9.18	9.42
	Hardness	mgCaCO3/L	1520	1480	1360	1290	1460	1880	1480	1650
	Al	mg/L	9.24	0.27	0.27	0.3	0.21	<0.005	<0.005	<0.005
	Sb	mg/L	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
	As	mg/L	<0.001	<0.001	0.001	<0.001	<0.001	<0.001	<0.001	<0.001
	Ba	mg/L	<0.001	<0.001	<0.001	0.009	<0.001	0.003	<0.001	<0.001
	Be	mg/L	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
	Bi	mg/L	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
	B	mg/L	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
	Cd	mg/L	0.022	0.0058	<0.0002	0.0017	0.012	<0.0002	<0.0002	<0.0002
	Ca	mg/L	487	492	541	511	500	476	519	499
	Cr	mg/L	0.1	0.006	0.005	0.003	<0.001	<0.001	0.002	<0.001
	Co	mg/L	4.19	0.21	0.14	0.33	3.75	<0.001	<0.001	<0.001
	Cu	mg/L	241	6.67	5.38	0.5	13.9	0.006	0.009	0.005
	Fe	mg/L	59.6	0.26	0.5	0.69	<0.05	<0.05	<0.05	<0.05
	Pb	mg/L	0.004	0.002	0.009	0.093	0.001	<0.001	<0.001	<0.001
	Li	mg/L	0.023	<0.001	<0.001	<0.001	0.015	0.008	<0.001	<0.001
	Mg	mg/L	74.1	61.1	1.8	2.85	50.7	168	44.7	97
	Mn	mg/L	1.29	0.055	0.026	0.046	0.82	<0.001	<0.001	<0.001
	Hg	ug/L	0.13	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
	Mo	mg/L	<0.0005	<0.0005	0.0005	<0.0005	0.0014	<0.0005	0.0074	0.013
	Ni	mg/L	103	3.95	3.09	6.84	79	0.004	0.015	0.008
	PO4	mg P/L	<0.15	<0.15	<0.15	<0.15	<0.15	<0.15	<0.15	<0.15
	K	mg/L	<0.1	1.4	<0.1	<0.1	0.3	2.6	0.4	0.7
	Se	mg/L	0.005	0.003	0.002	<0.001	0.002	0.003	0.01	0.024
	Si	mg SiO2/L	0.8	0.8	<0.25	0.5	1.4	<0.25	15.8	10.5
	Ag	mg/L	0.0064	0.0077	<0.00025	<0.00025	0.0012	<0.00025	<0.00025	<0.00025
	Na	mg/L	47.2	147	2.8	4.63	41.9	238	51.4	97.6
	Sr	mg/L	0.26	0.32	0.16	0.3	0.19	0.24	0.18	0.26
	Te	mg/L	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
	Tl	mg/L	0.0006	<0.0001	0.0002	0.0001	0.0012	<0.0001	<0.0001	<0.0001
	Th	mg/L	0.033	0.01	<0.0005	<0.0005	<0.0005	<0.0005	<0.0005	<0.0005
Sn	mg/L	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	
Ti	mg/L	0.044	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	
U	mg/L	0.0017	<0.0005	<0.0005	<0.0005	<0.0005	<0.0005	<0.0005	<0.0005	
V	mg/L	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	
Zn	mg/L	5.85	0.17	0.095	0.24	4.4	<0.005	<0.005	<0.005	
Zr	mg/L	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	

Notes
#N/A - result checking in progress

Procedure	Parameter	Unit	Residues							
			Leach, no CuSO4	Leach, with CuSO4	Gypsum	Raffinate Neutralization	Fe/Al	Mg	Combined	Combined no Gypsum
TCLP Leachate Extraction Results	pH	-	4.74	4.94	4.95	4.96	4.98	9.8	5.23	8.98
	Hardness	mgCaCO3/L	1500	1260	1490	1900	1610	3270	2400	3710
	Al	mg/L	2.6	0.008	0.22	0.28	1.51	<0.005	0.088	<0.005
	Sb	mg/L	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
	As	mg/L	<0.001	<0.001	0.001	0.001	<0.001	<0.001	<0.001	<0.001
	Ba	mg/L	<0.001	0.001	0.001	0.014	0.001	0.004	0.007	0.003
	Be	mg/L	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
	Bi	mg/L	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
	B	mg/L	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
	Cd	mg/L	0.021	0.0055	<0.0002	0.0018	0.011	<0.0002	0.0025	<0.0002
	Ca	mg/L	451	402	594	754	562	586	733	718
	Cr	mg/L	0.031	0.002	0.006	0.007	0.13	0.002	0.022	0.004
	Co	mg/L	4.48	0.21	0.14	0.32	3.37	0.001	0.096	0.001
	Cu	mg/L	244	5.75	5.28	0.46	46.8	0.018	2.41	0.005
	Fe	mg/L	3.9	<0.05	0.42	0.55	0.06	<0.05	0.14	<0.05
	Pb	mg/L	0.001	<0.001	0.012	0.34	<0.001	<0.001	0.004	<0.001
	Li	mg/L	0.025	0.002	0.001	0.002	0.014	0.008	<0.001	0.002
	Mg	mg/L	91	62	2	2.74	50	438	137	464
	Mn	mg/L	1.39	0.057	0.029	0.046	0.76	<0.001	0.11	<0.001
	Hg	ug/L	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
	Mo	mg/L	<0.0005	<0.0005	0.0007	0.0006	0.0017	<0.0005	0.0006	0.027
	Ni	mg/L	107	4	3.15	6.35	73.4	0.018	3.02	0.093
	P	mg PO4/L	<0.15	<0.15	<0.15	1	<0.15	<0.15	<0.15	<0.15
	K	mg/L	0.6	2.2	0.5	0.6	0.7	3	1.2	1.7
	Se	mg/L	0.004	0.003	0.002	0.002	0.002	0.002	0.002	0.033
	Si	mg SiO2/L	3.5	4	1.1	1.5	2.7	0.8	14.9	14.1
	Ag	mg/L	0.0074	0.0093	<0.00025	<0.00025	0.0015	<0.00025	<0.00025	<0.00025
	Na	mg/L	1730	1680	1560	1600	1540	1710	1590	1710
	Sr	mg/L	0.29	0.35	0.22	0.46	0.29	0.41	0.34	0.47
	Te	mg/L	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
	Tl	mg/L	0.0009	<0.0001	0.0002	0.0003	0.0016	<0.0001	0.0003	0.0001
	Th	mg/L	0.002	0.0013	<0.0005	<0.0005	<0.0005	<0.0005	<0.0005	<0.0005
Sn	mg/L	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	0.001	<0.001	
Ti	mg/L	0.009	<0.001	<0.001	<0.001	<0.001	<0.001	0.001	<0.001	
U	mg/L	0.0016	<0.0005	<0.0005	<0.0005	0.0069	<0.0005	0.0008	<0.0005	
V	mg/L	0.003	0.003	0.003	0.003	0.001	0.003	0.002	0.003	
Zn	mg/L	5.49	0.18	0.12	0.25	4.12	<0.005	0.22	<0.005	
Zr	mg/L	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	

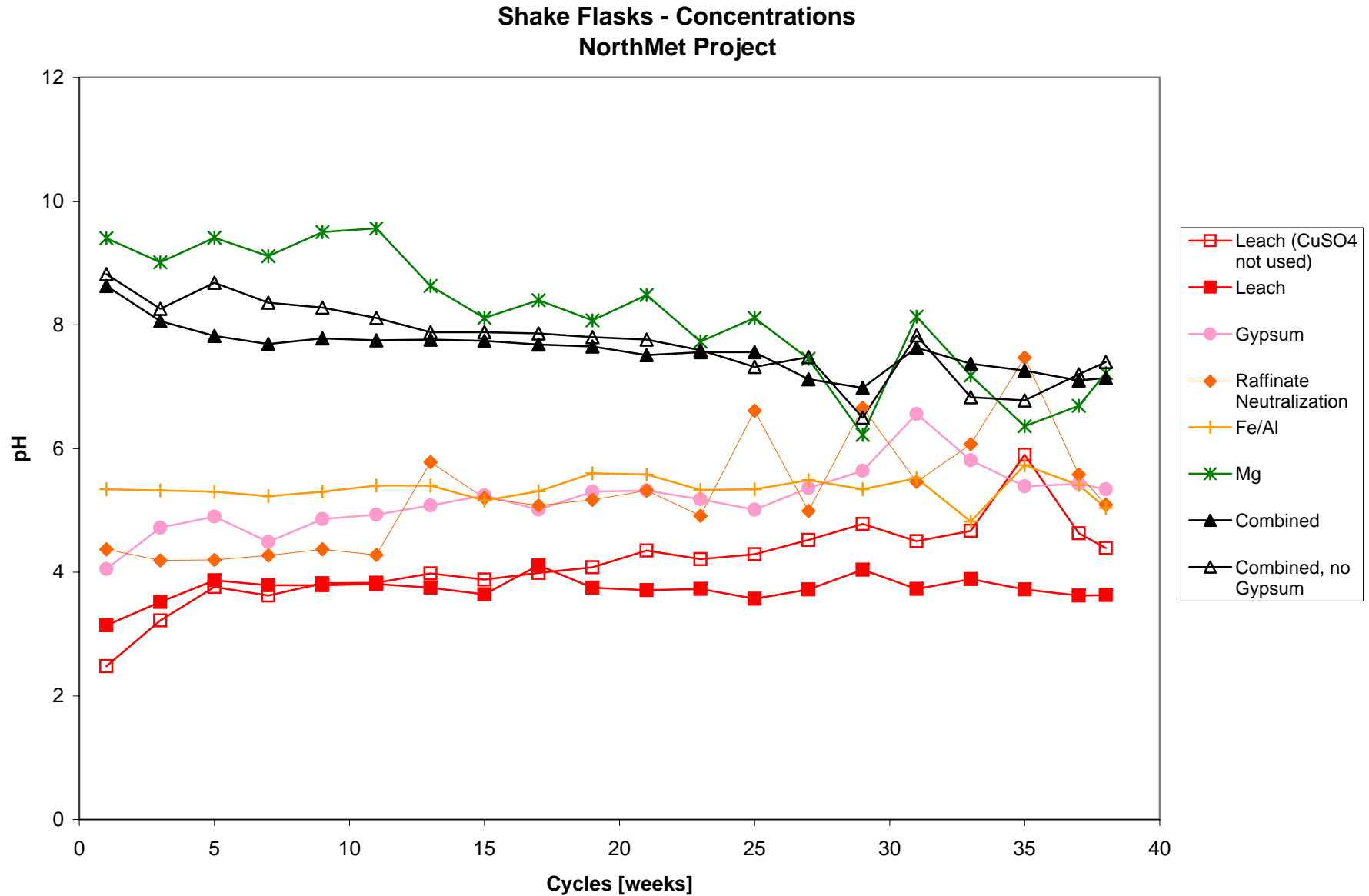
Notes
#N/A - result checking in progress

Procedure	Parameter	Unit	Residues							
			Leach, no CuSO4	Leach, with CuSO4	Gypsum	Raffinate Neutralization	Fe/Al	Mg	Combined	Combined no Gypsum
3:1 Water Leach (First Week)	pH	-	2.12	2.64	3.30	4.22	5.06	9.33	8.65	8.95
	ORP	mV	625	601	568	530	490	277	390	262
	Cond.	µS/cm	12500	9720	13	2490	6290	14530	4480	7690
	Acidity to pH 4.5	mg CaCO3/L	1807.5	252.0	38.0	2.25	#N/A	#N/A	#N/A	#N/A
	Total Acidity	mg CaCO3/L	4362.5	365.0	119.0	58.5	400.0	#N/A	#N/A	#N/A
	Alkalinity	mg CaCO3/L	#N/A	#N/A	#N/A	#N/A	5.0	52.0	39.5	46.0
	Inorganic C	mg CaCO3/L	<1	<1	<1	<1	<1	7	9	8
	Hardness	mg CaCO3/L	2640	2810	1360	1300	3000	6020	2000	3730
	F	mg/L	<1	<0.5	<0.25	3.6	1.6	<1	<5	<0.5
	Cl	mg/L	717	717	21.7	23.5	473	1950	392	635
	SO4	mg/L	9485	4507	1600	1670	4490	7940	2430	4280
	Al	mg/L	47.9	1.34	1.35	1.69	1.06	<0.005	<0.005	<0.005
	Sb	mg/L	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
	As	mg/L	0.012	<0.001	0.004	0.003	0.002	<0.001	<0.001	<0.001
	Ba	mg/L	<0.001	<0.001	0.006	0.012	0.002	0.005	0.001	0.002
	Be	mg/L	0.003	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
	Bi	mg/L	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
	B	mg/L	0.07	<0.05	<0.05	<0.05	0.11	<0.05	<0.05	<0.05
	Cd	mg/L	0.102	0.031	0.0035	0.0082	0.064	<0.0002	<0.0002	<0.0002
	Ca	mg/L	322	434	522	494	463	458	450	453
	Cr	mg/L	0.54	0.03	0.02	0.013	0.001	0.001	0.006	0.002
	Co	mg/L	30.2	0.99	0.79	1.8	29.1	0.001	<0.001	<0.001
	Cu	mg/L	1300	51.1	31.4	2.81	52	0.025	0.002	0.006
	Fe	mg/L	511	12.6	2.72	3.94	0.12	<0.05	<0.05	<0.05
	Pb	mg/L	0.009	0.006	0.018	0.3	0.002	<0.001	<0.001	<0.001
	Li	mg/L	0.14	0.005	0.003	0.005	0.096	0.048	<0.001	0.001
	Mg	mg/L	446	419	10.1	15.9	446	955	213	630
	Mn	mg/L	6.69	0.28	0.13	0.28	4.83	<0.001	<0.001	<0.001
	Hg	mg/L	1.18	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
	Mo	mg/L	<0.0005	<0.0005	0.0013	<0.0005	0.0023	<0.0005	0.027	0.03
	Ni	mg/L	616	19	15.7	37.9	616	0.034	0.028	0.054
	P	mg/L	0.7	<0.15	<0.15	<0.15	<0.15	<0.15	<0.15	<0.15
	K	mg/L	0.4	7.6	0.1	0.5	2.2	20.3	1.8	3.9
	Se	mg/L	0.01	0.003	<0.001	0.001	0.003	<0.001	0.011	0.021
	Si	mg/L	3.3	3.2	1.2	2.7	4.5	<0.25	14.8	5.7
	Ag	mg/L	0.022	0.025	0.0003	<0.00025	0.013	<0.00025	<0.00025	<0.00025
	Na	mg/L	290	616	16	20.9	284	2020	255	717
	Sr	mg/L	0.17	0.38	0.2	0.46	0.22	0.3	0.2	0.27
	Te	mg/L	<0.001	<0.001	0.001	<0.001	<0.001	<0.001	<0.001	<0.001
	Tl	mg/L	0.0029	0.0005	0.0006	0.0005	0.0049	0.0004	<0.0001	0.0001
Th	mg/L	0.145	0.109	0.0007	<0.0005	<0.0005	<0.0005	<0.0005	<0.0005	
Sn	mg/L	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	
Ti	mg/L	0.42	0.005	0.002	0.001	0.001	<0.001	<0.001	<0.001	
U	mg/L	0.0081	<0.0005	<0.0005	0.0009	0.001	<0.0005	<0.0005	<0.0005	
V	mg/L	0.12	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	
Zn	mg/L	48.6	0.92	0.15	1.34	34.7	<0.005	<0.005	<0.005	
Zr	mg/L	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	

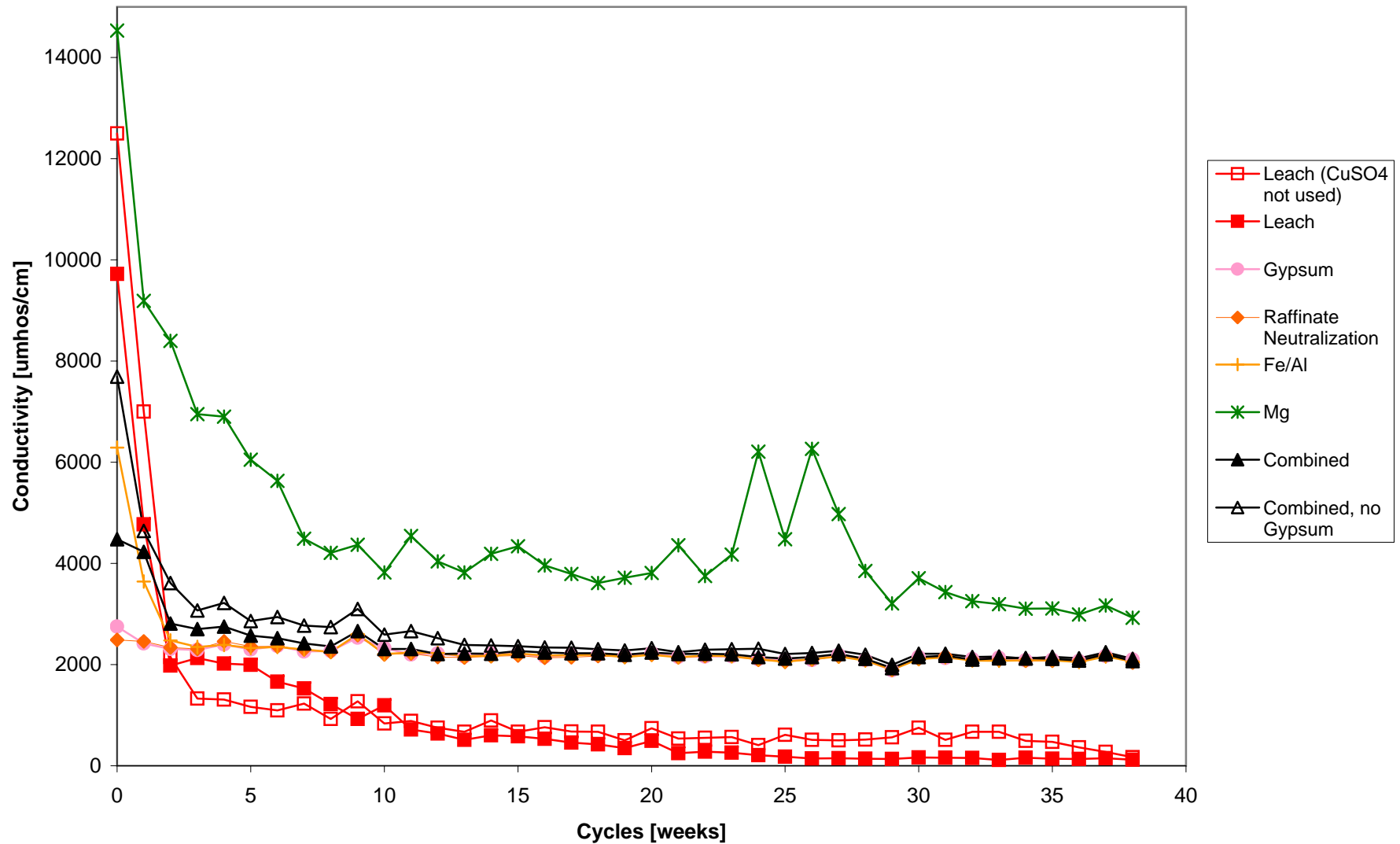
Notes
#N/A - result checking in progress

Appendix C
Leachate Chemistry Graphs

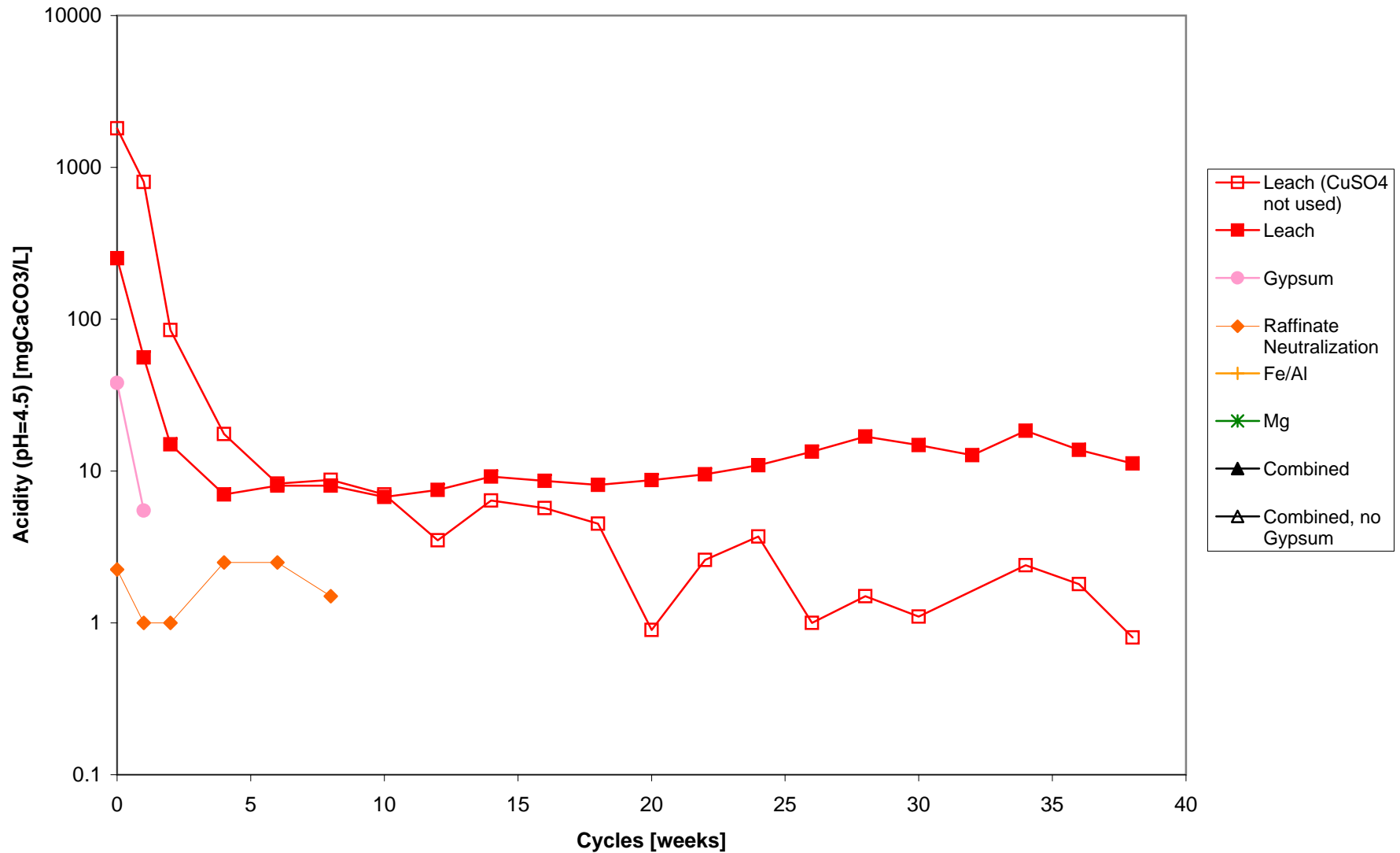
Appendix C.1
Sequential Shake Flasks



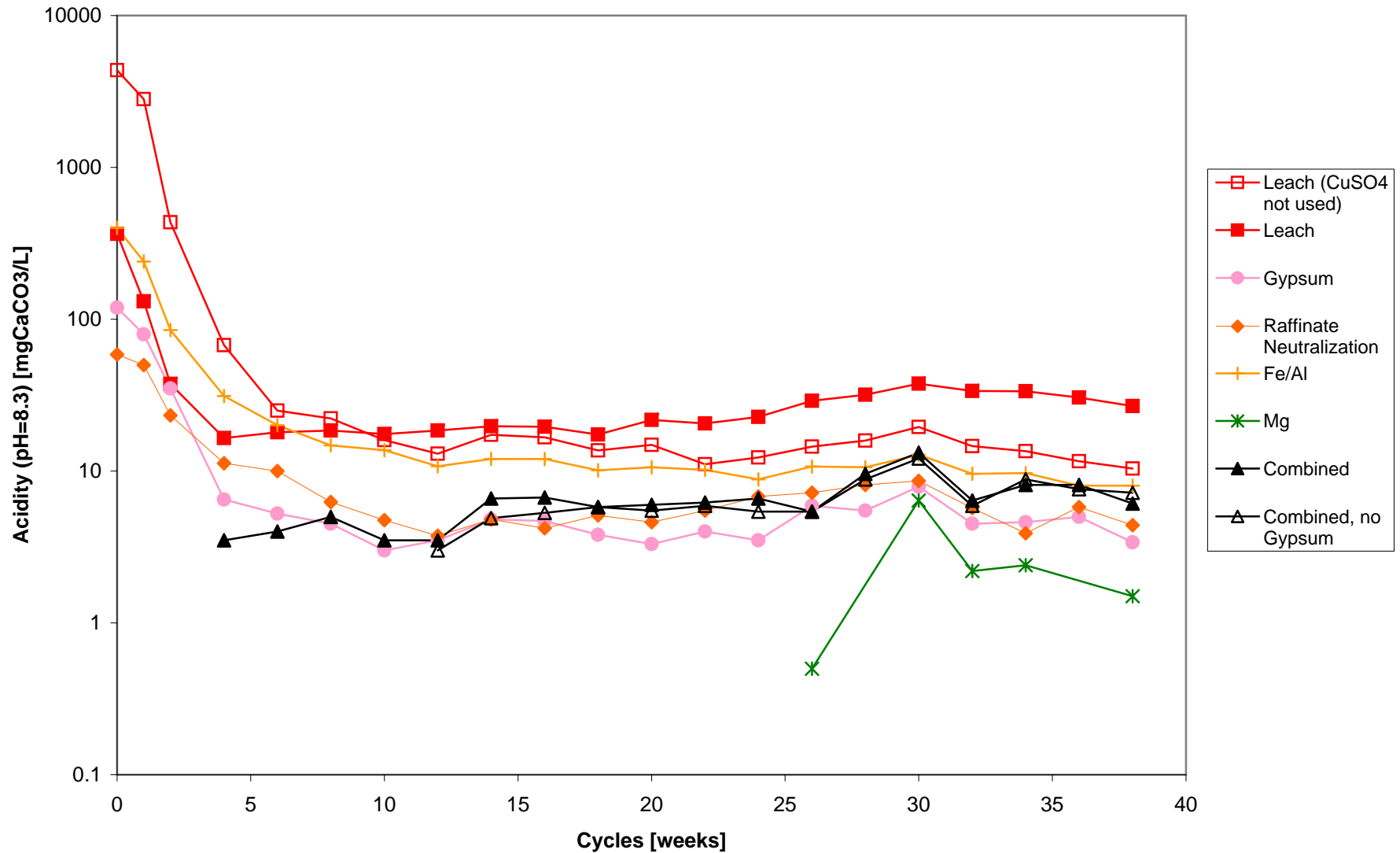
Shake Flasks - Concentrations
NorthMet Project



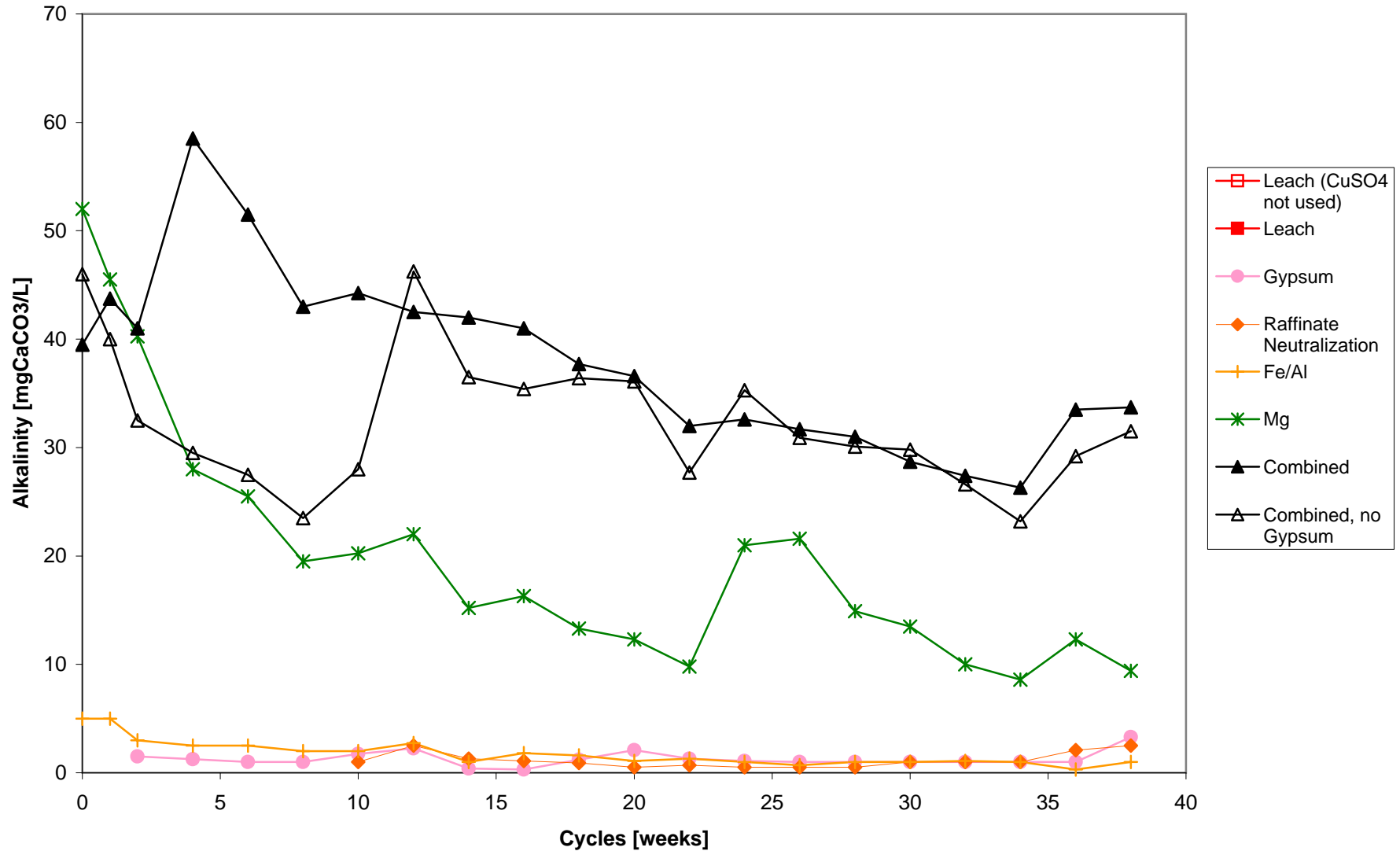
Shake Flasks - Concentrations
NorthMet Project

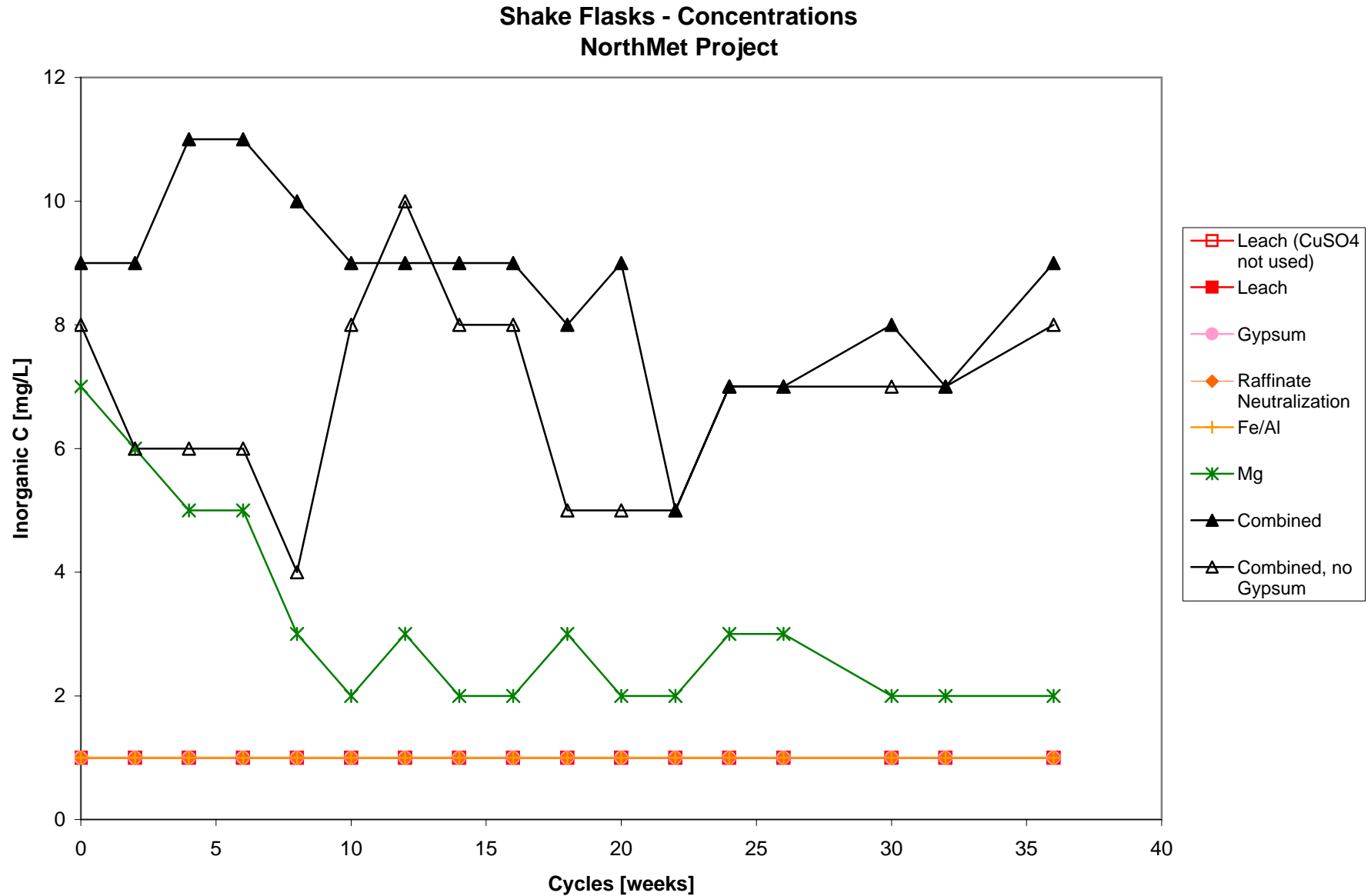


Shake Flasks - Concentrations
NorthMet Project

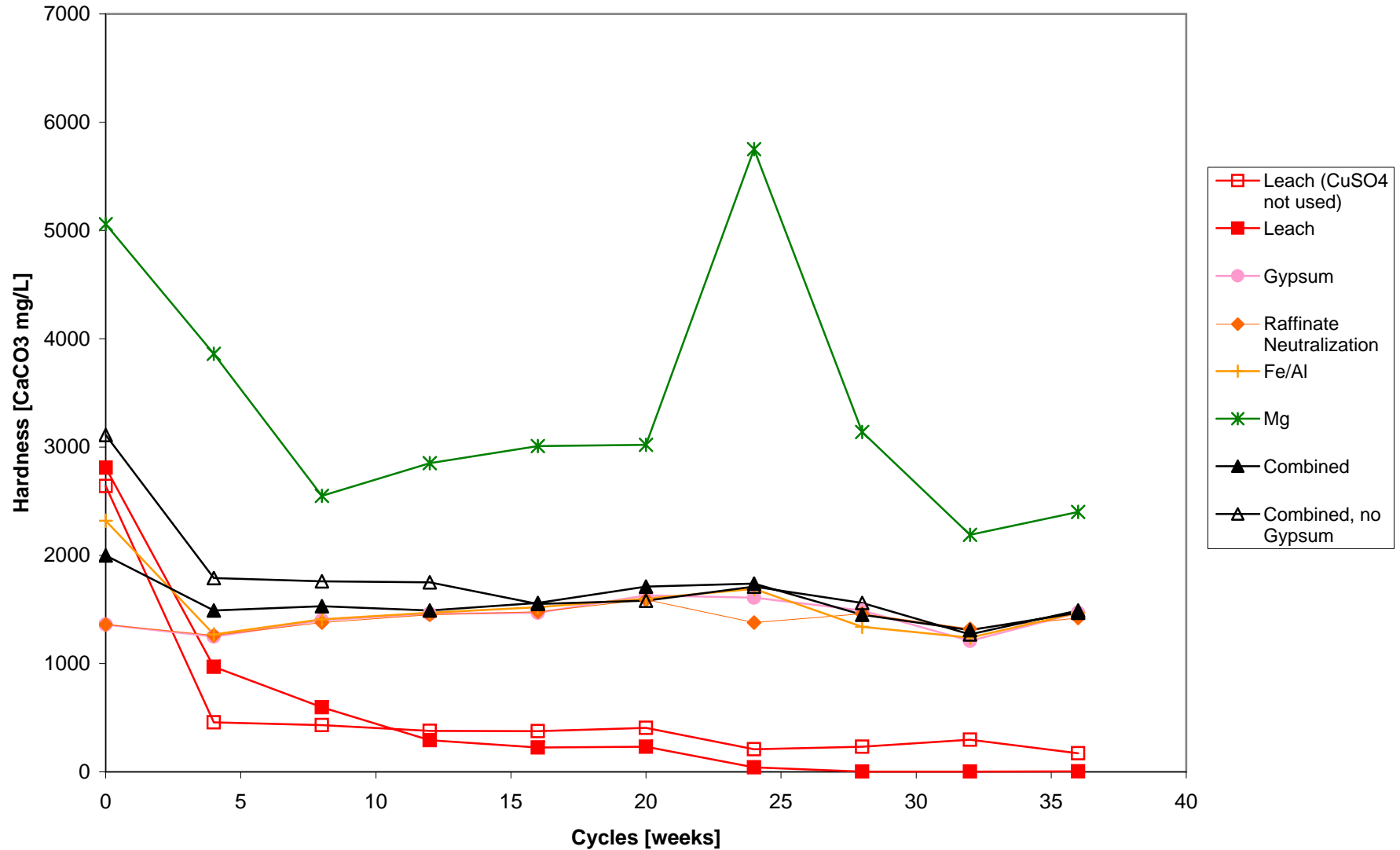


Shake Flasks - Concentrations
NorthMet Project

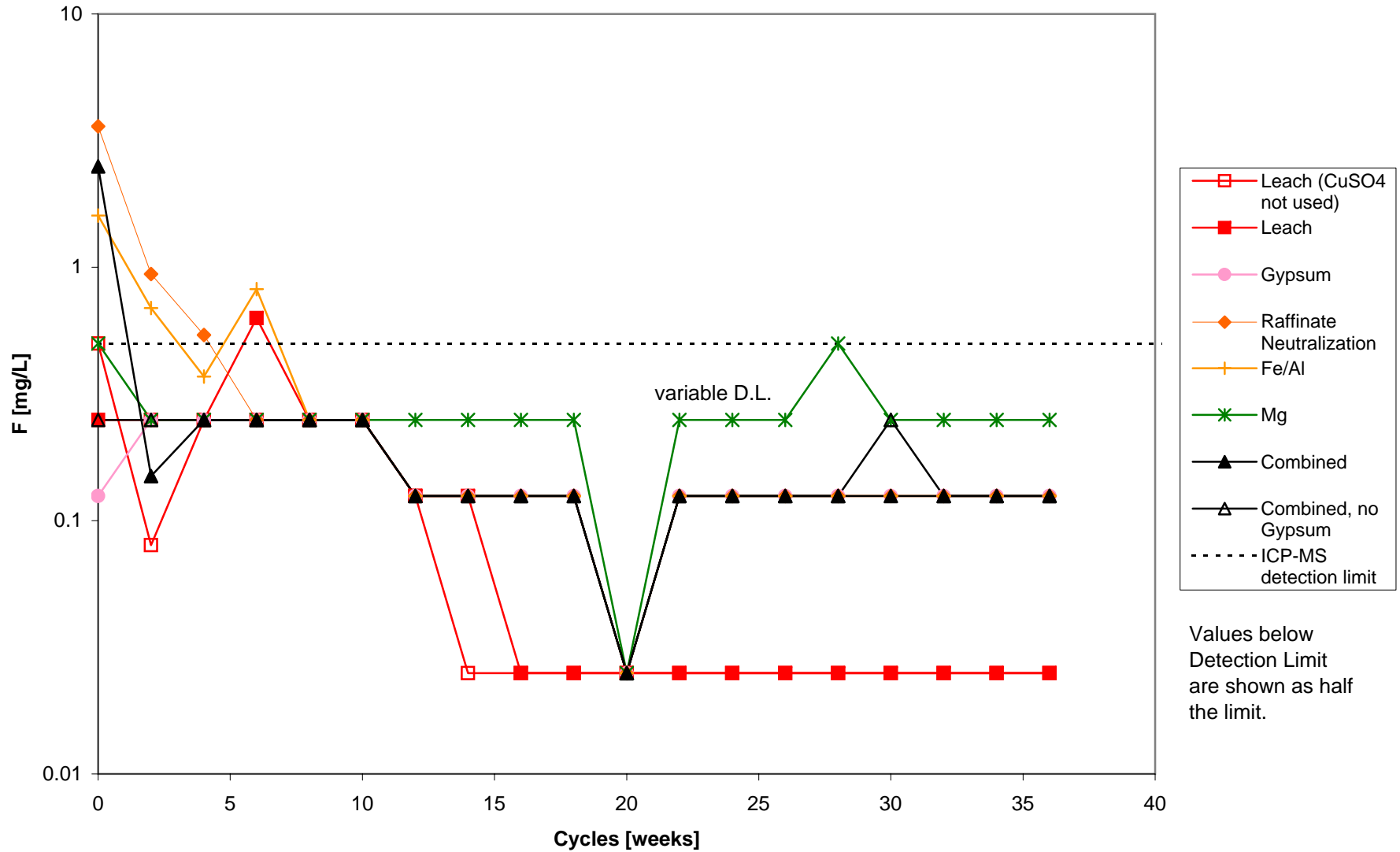




Shake Flasks - Concentrations
NorthMet Project

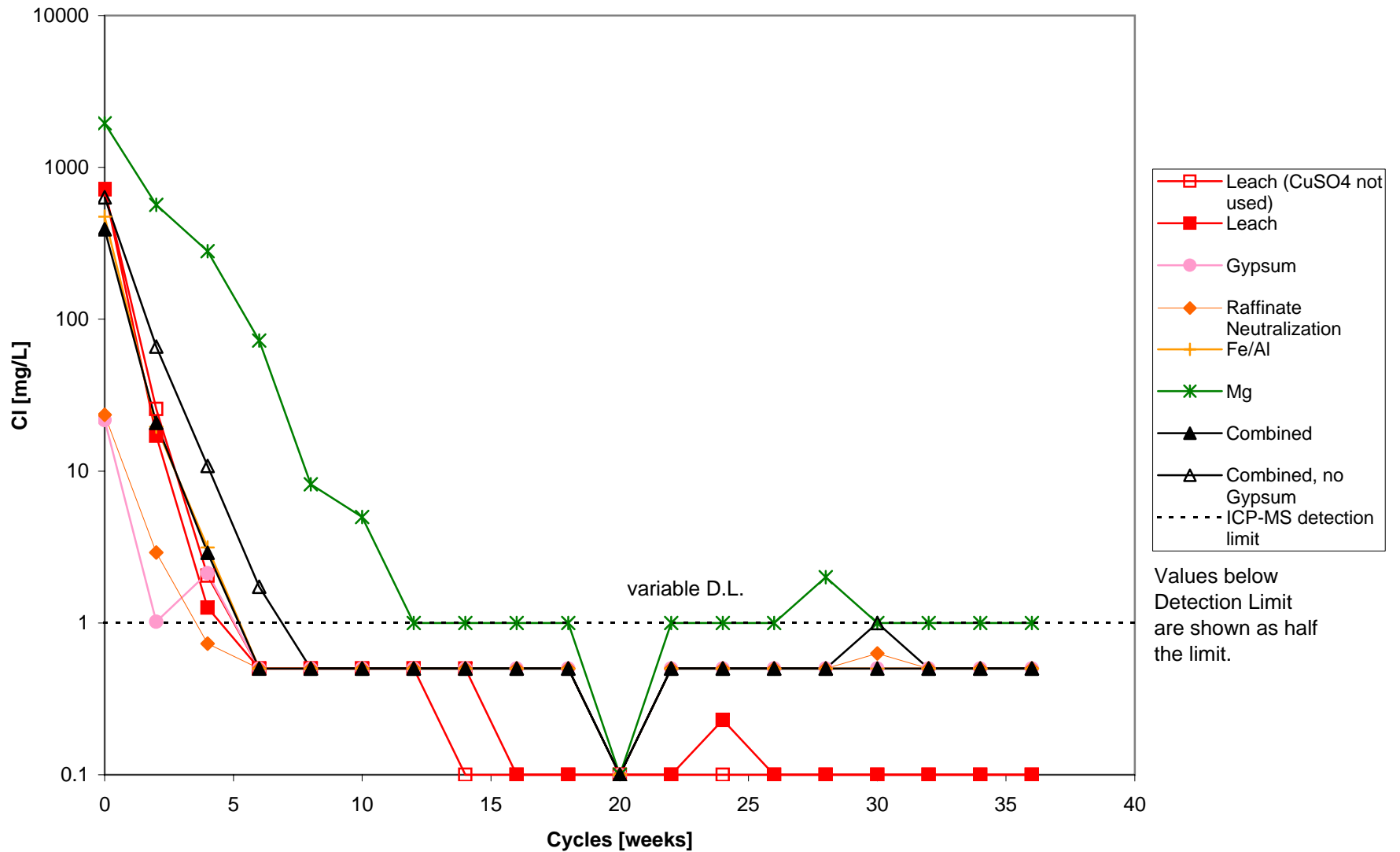


Shake Flasks - Concentrations
NorthMet Project

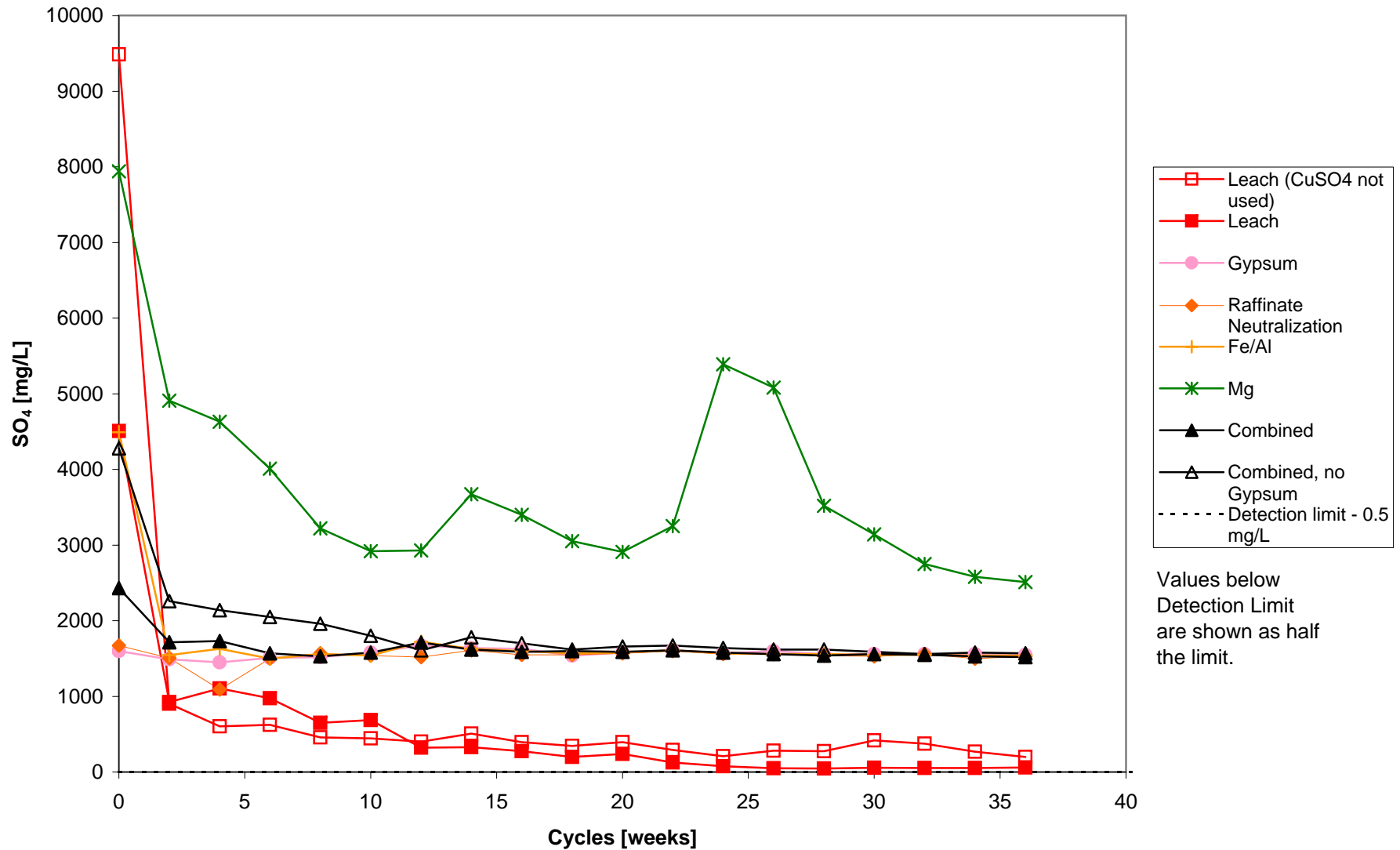


Values below
Detection Limit
are shown as half
the limit.

Shake Flasks - Concentrations
NorthMet Project

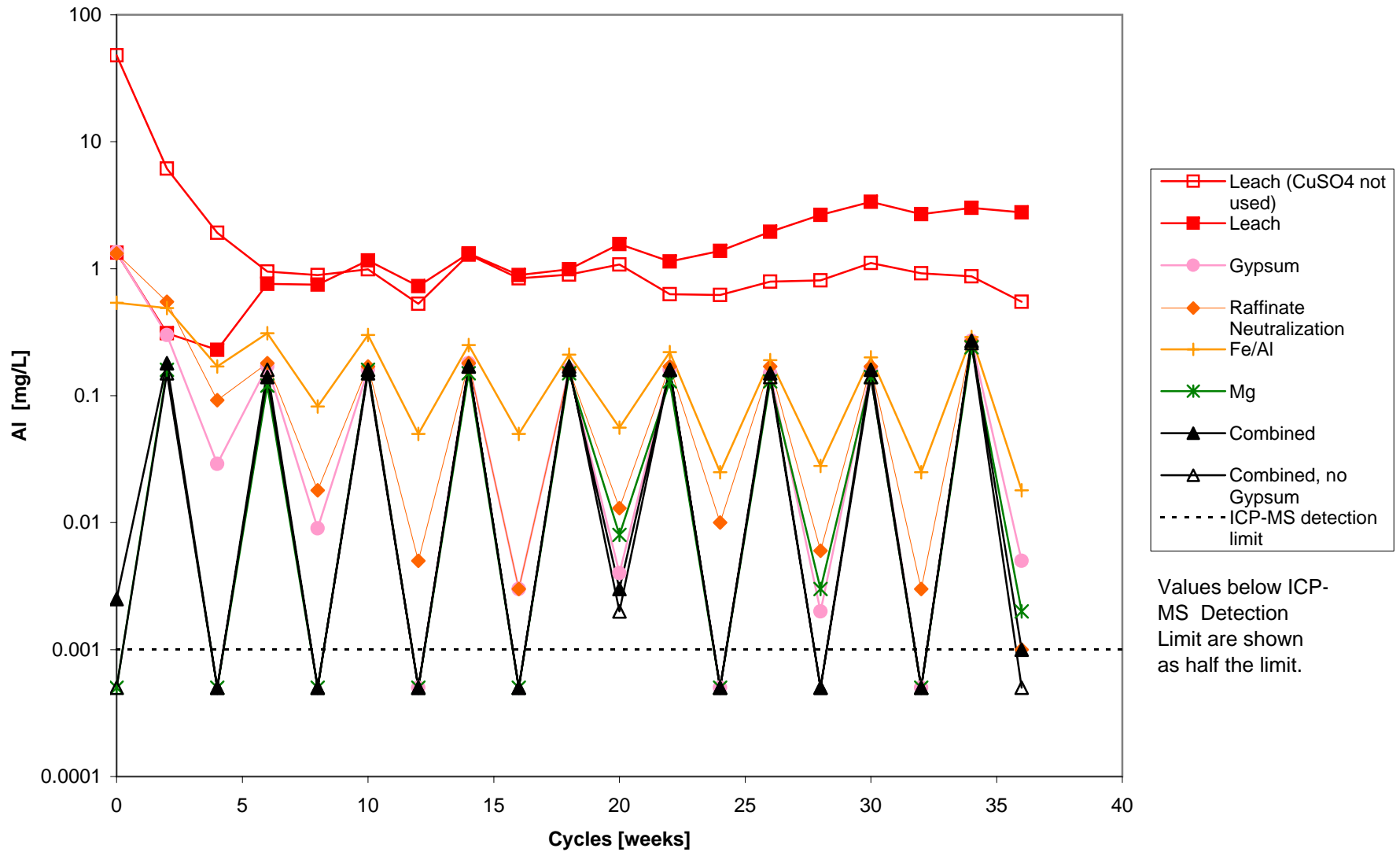


Shake Flasks - Concentrations
NorthMet Project

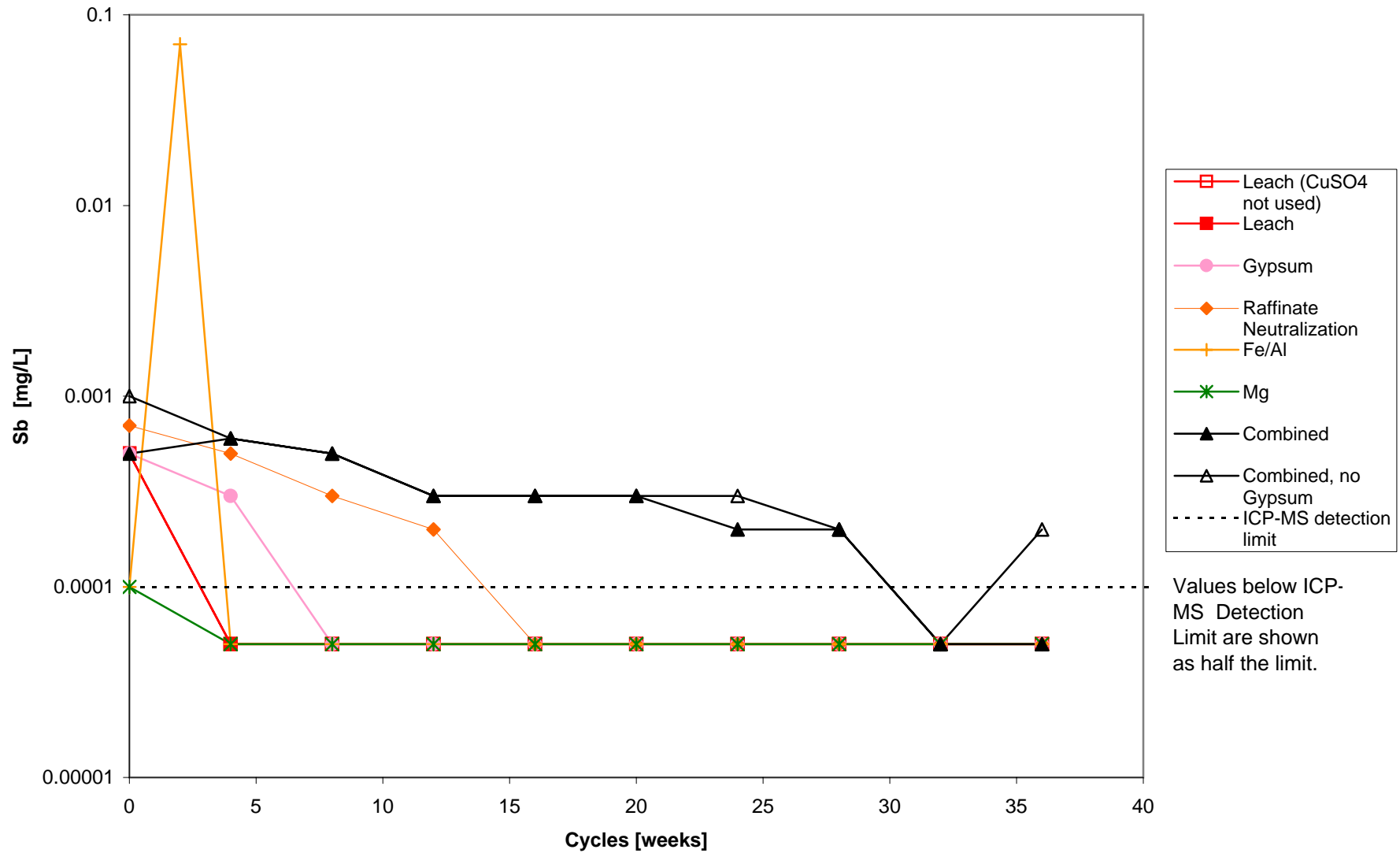


Values below
Detection Limit
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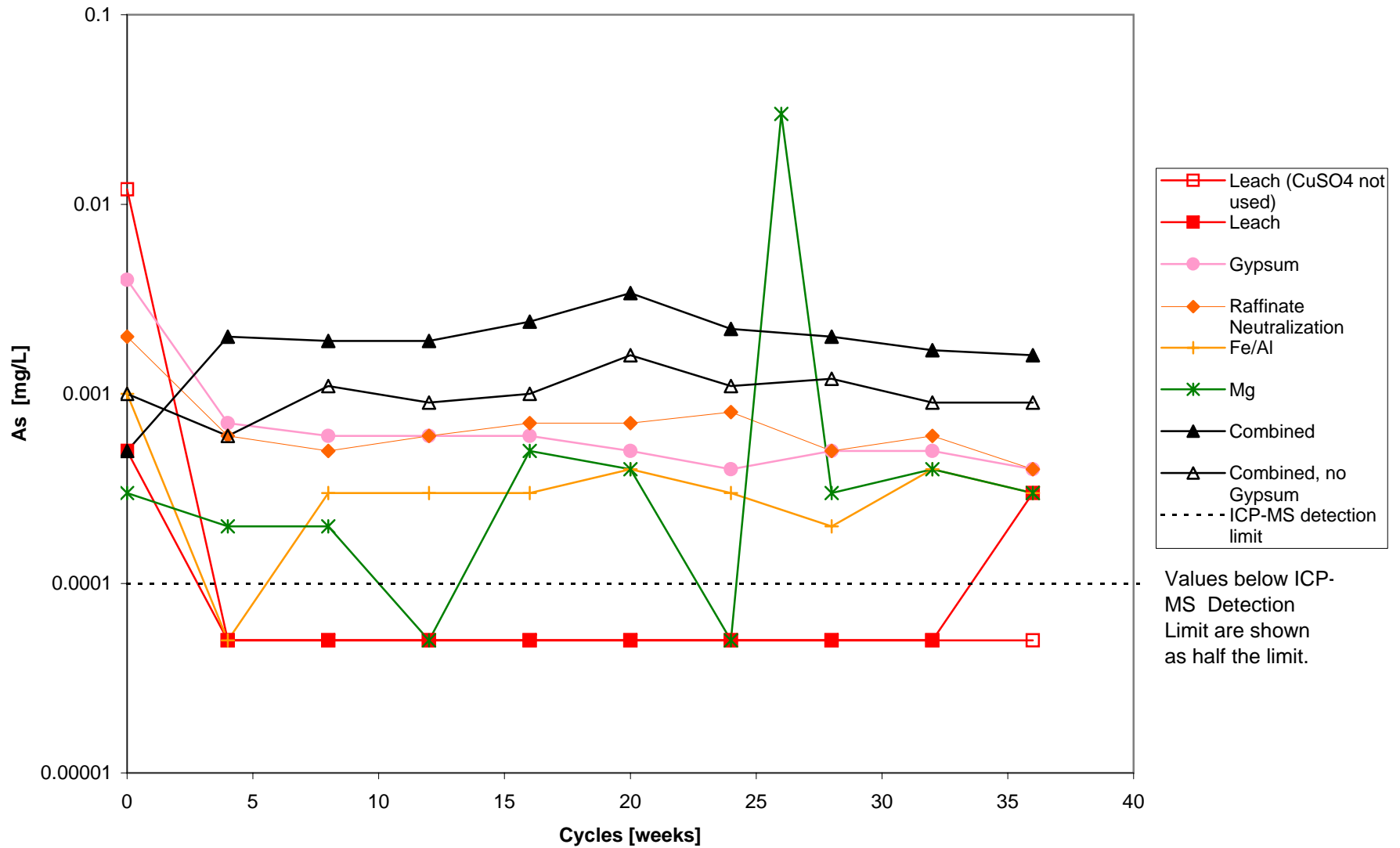
Shake Flasks - Concentrations
NorthMet Project



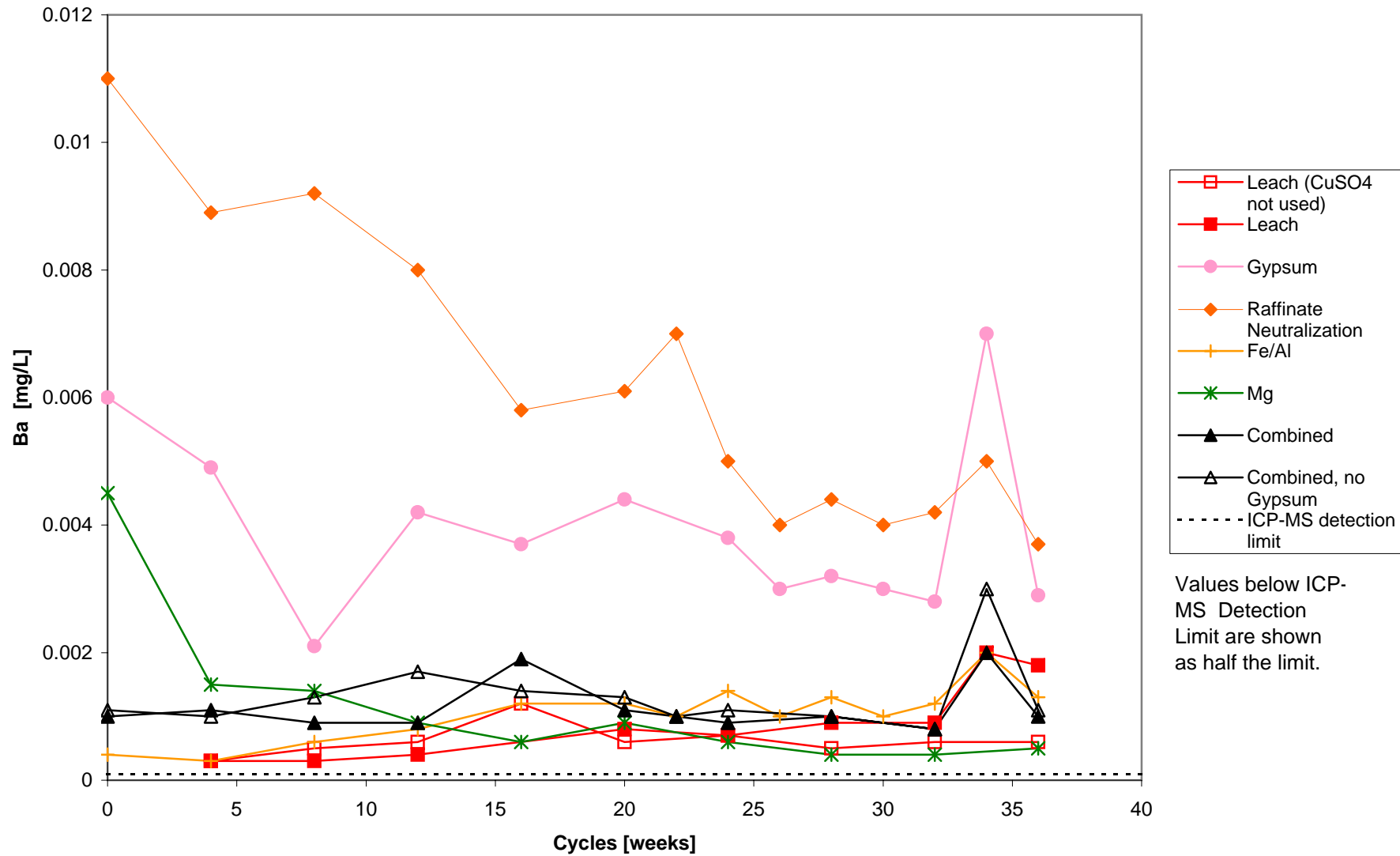
Shake Flasks - Concentrations
NorthMet Project



Shake Flasks - Concentrations
NorthMet Project

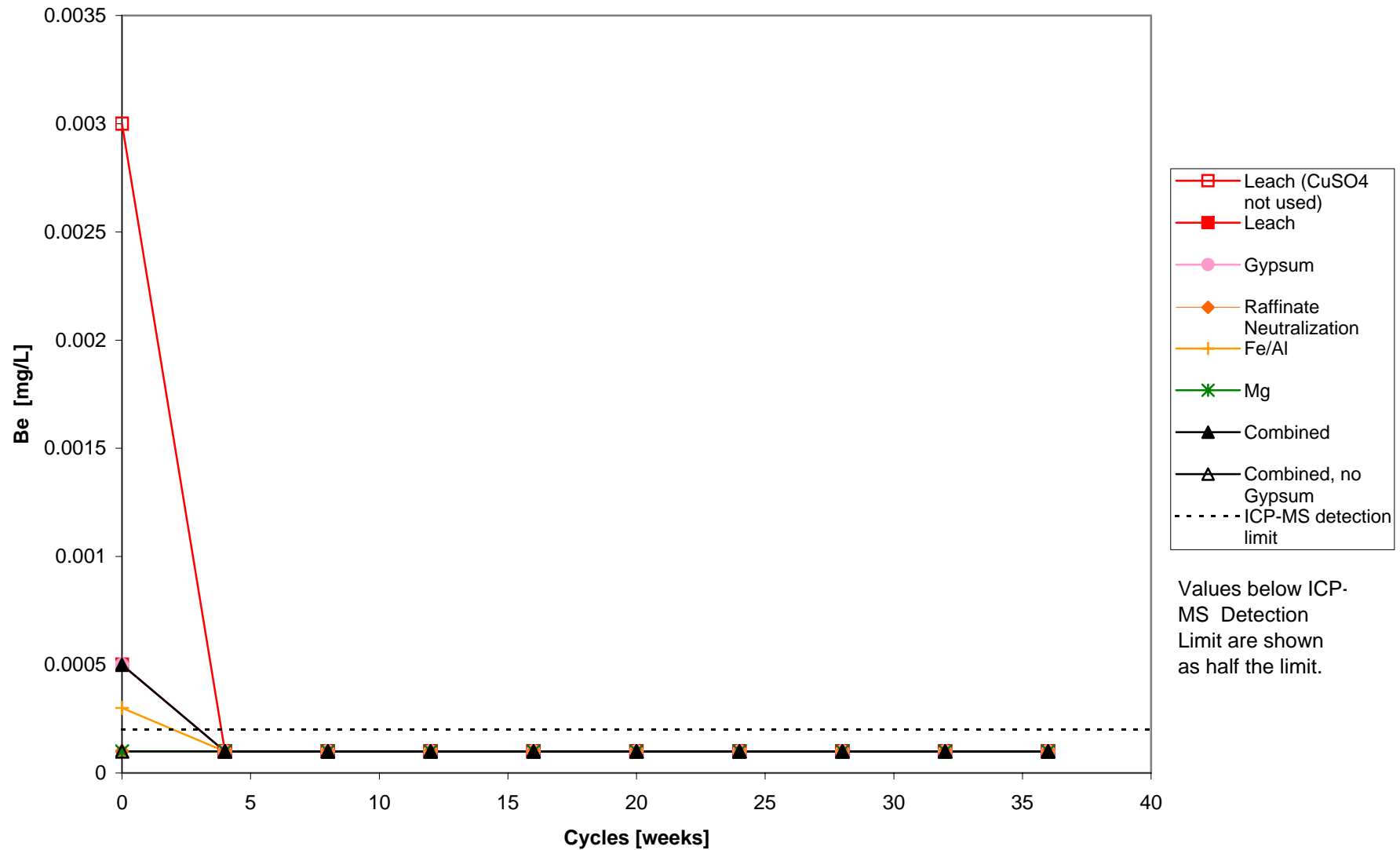


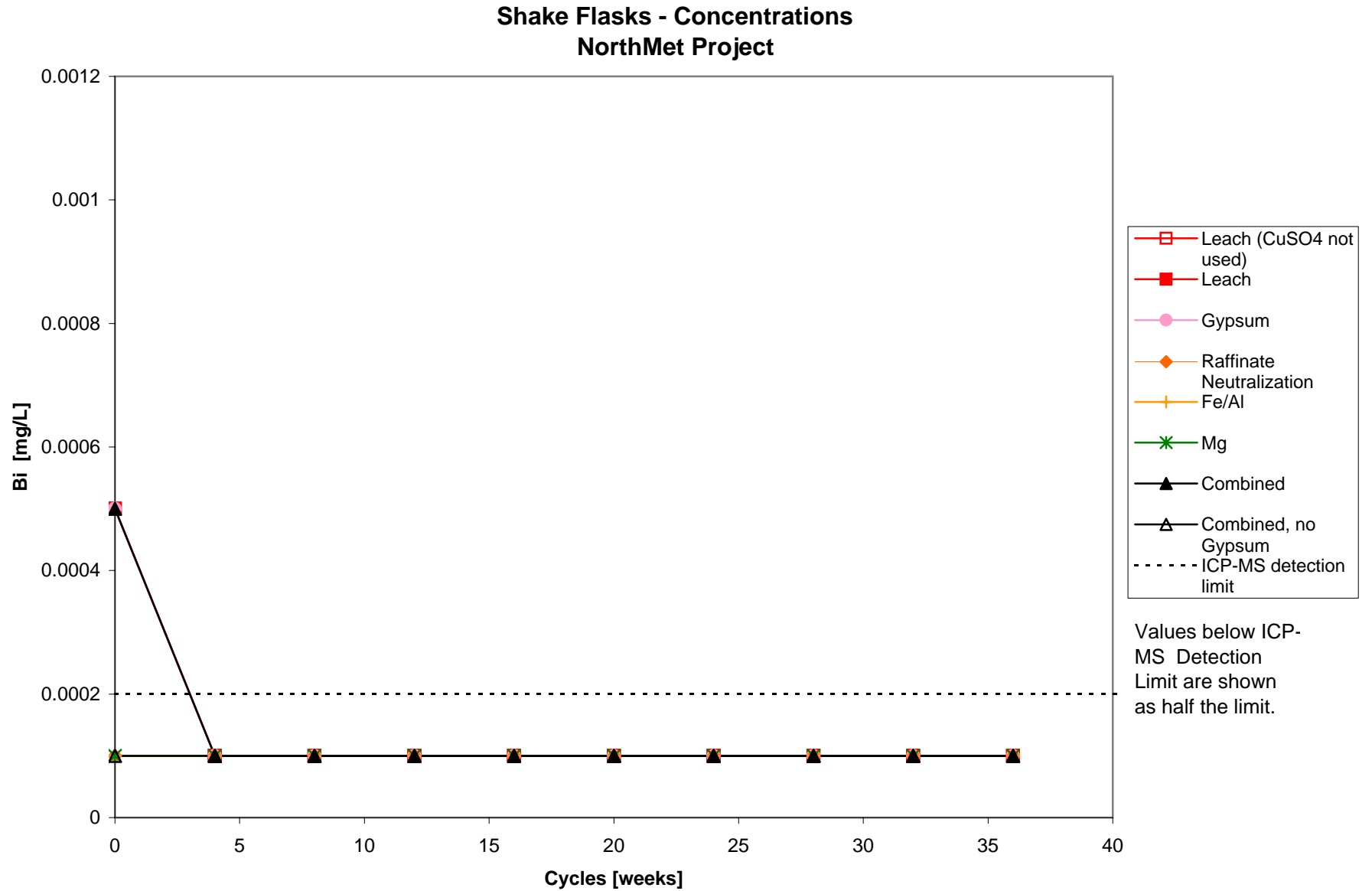
Shake Flasks - Concentrations
NorthMet Project



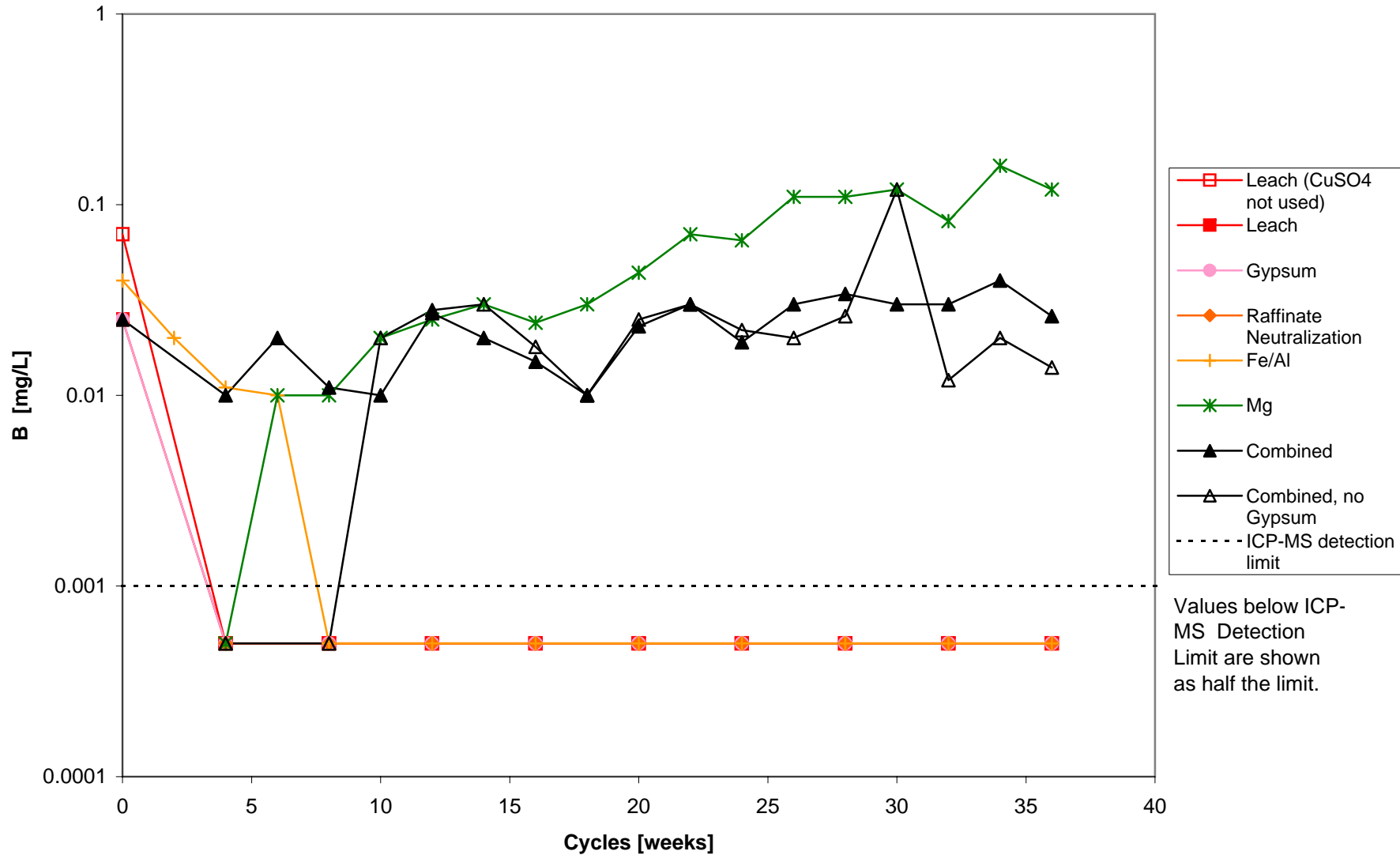
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Shake Flasks - Concentrations
NorthMet Project



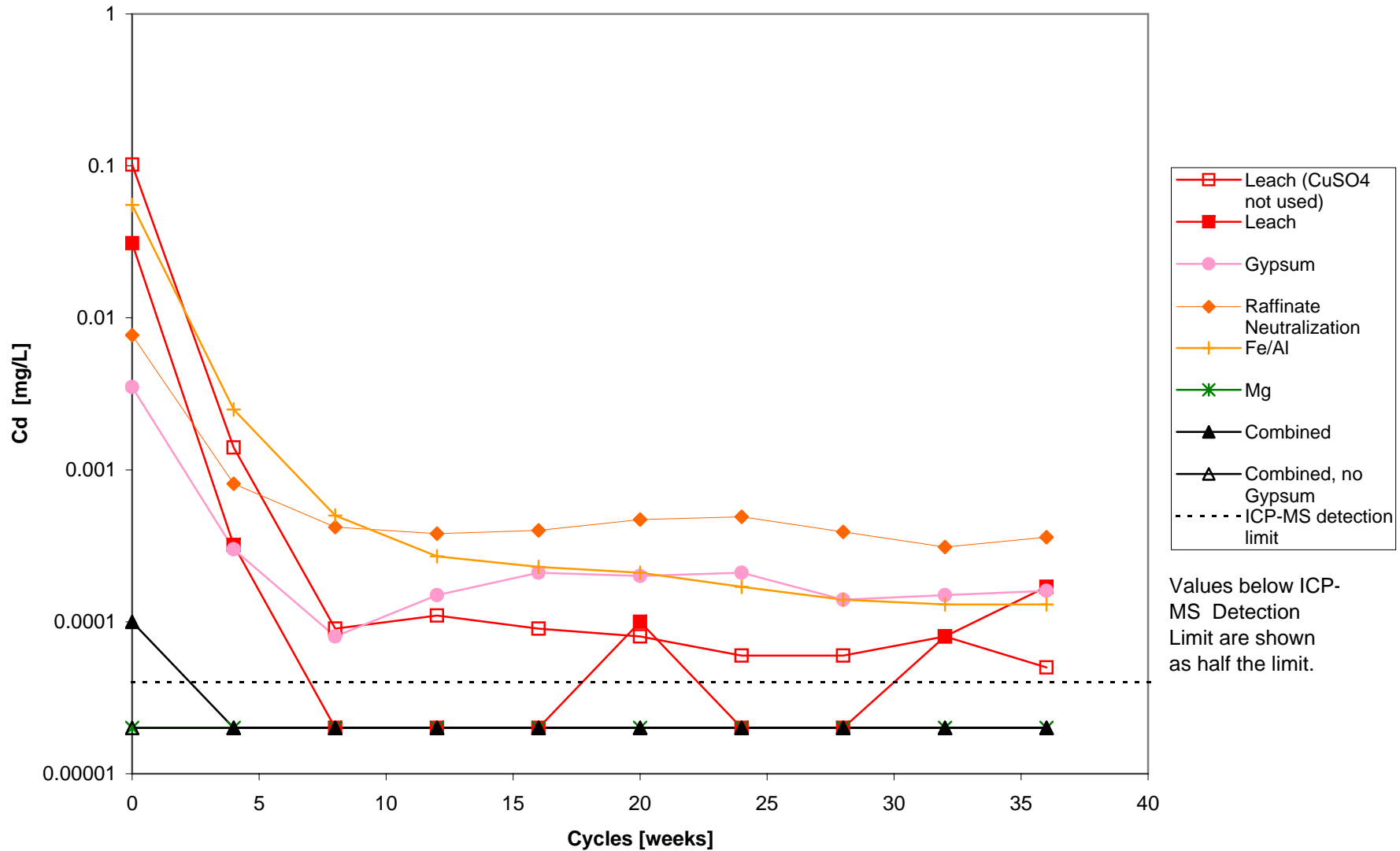


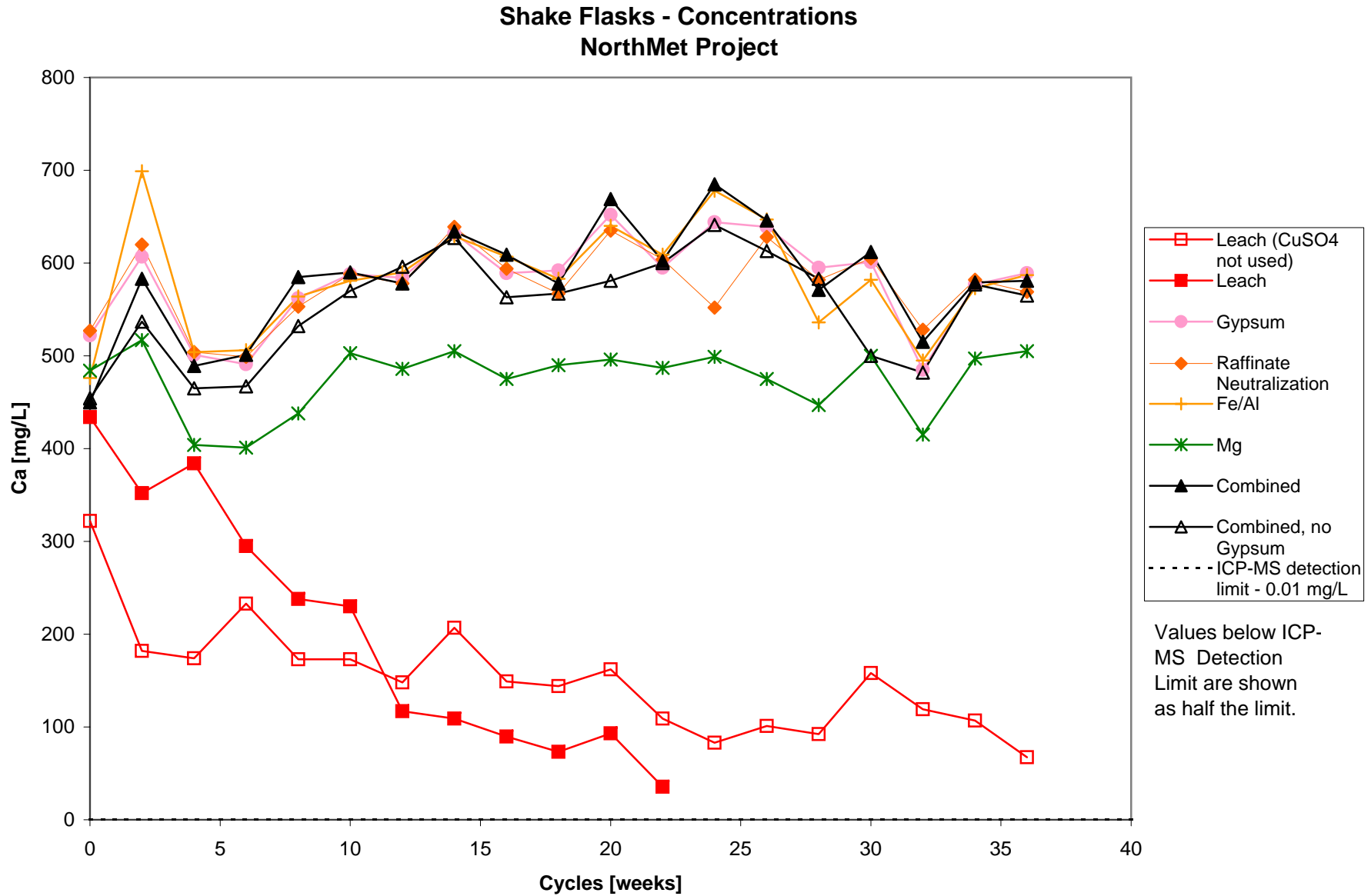
Shake Flasks - Concentrations
NorthMet Project



Values below ICP-MS Detection Limit are shown as half the limit.

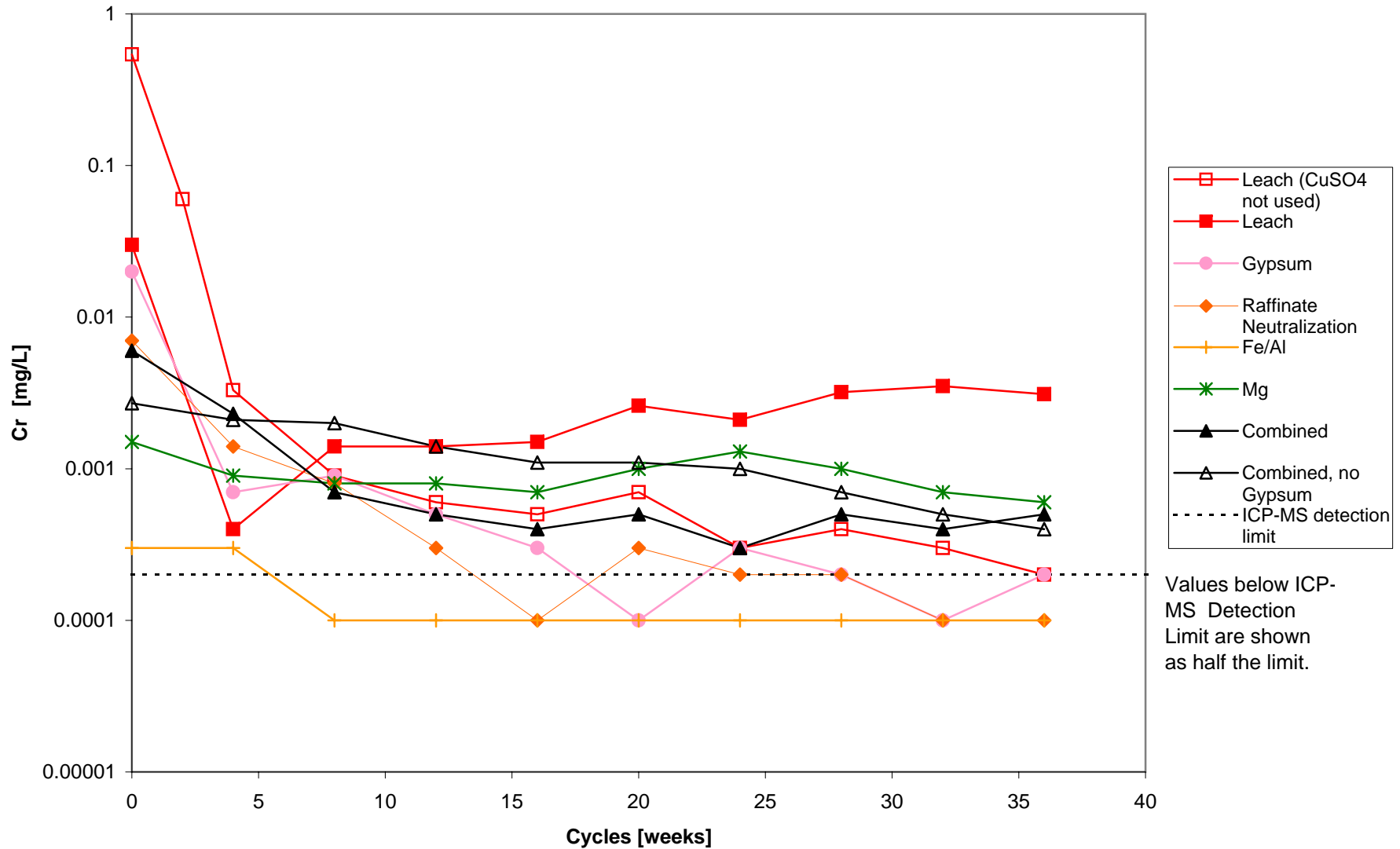
Shake Flasks - Concentrations
NorthMet Project





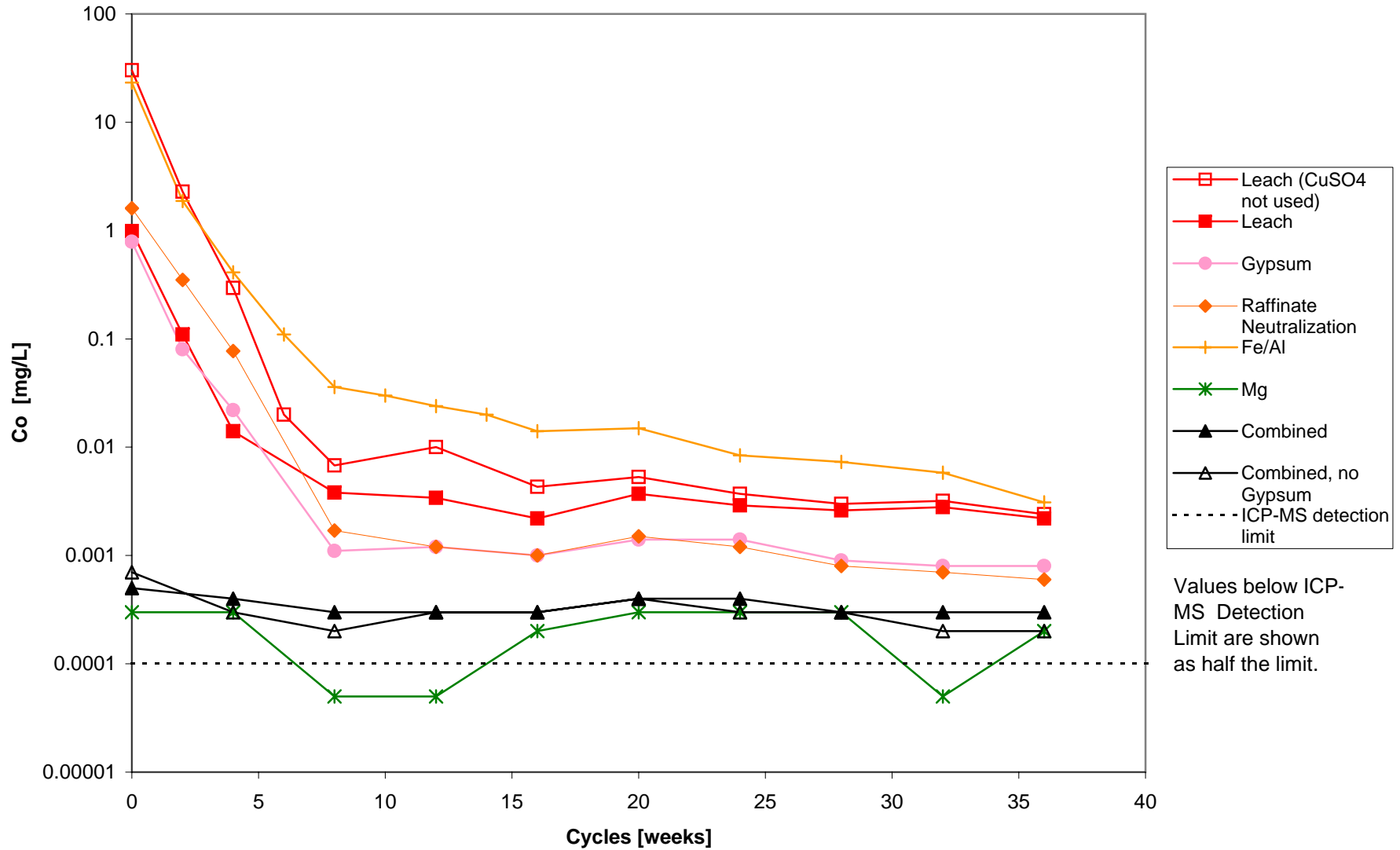
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Shake Flasks - Concentrations
NorthMet Project

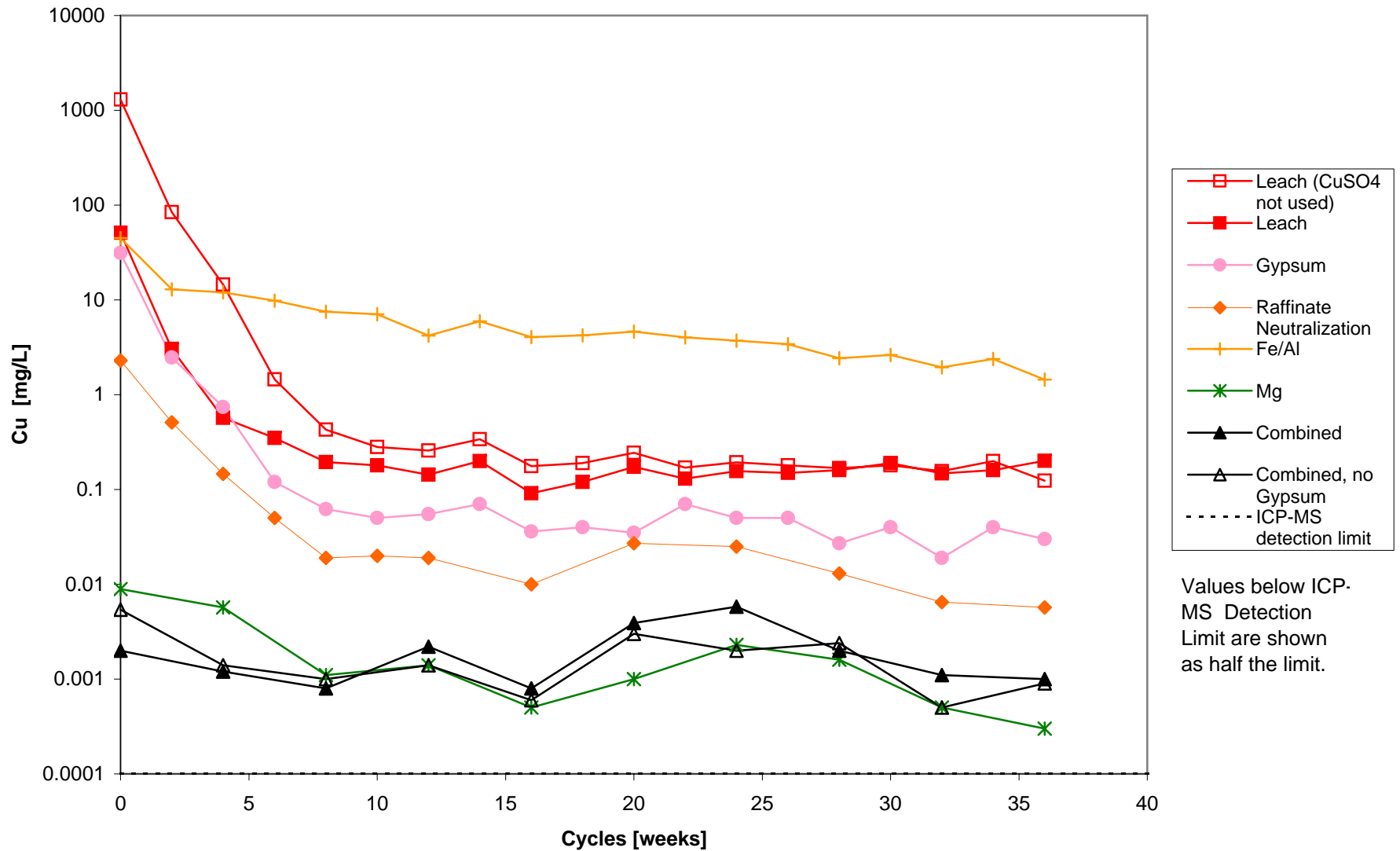


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Shake Flasks - Concentrations
NorthMet Project

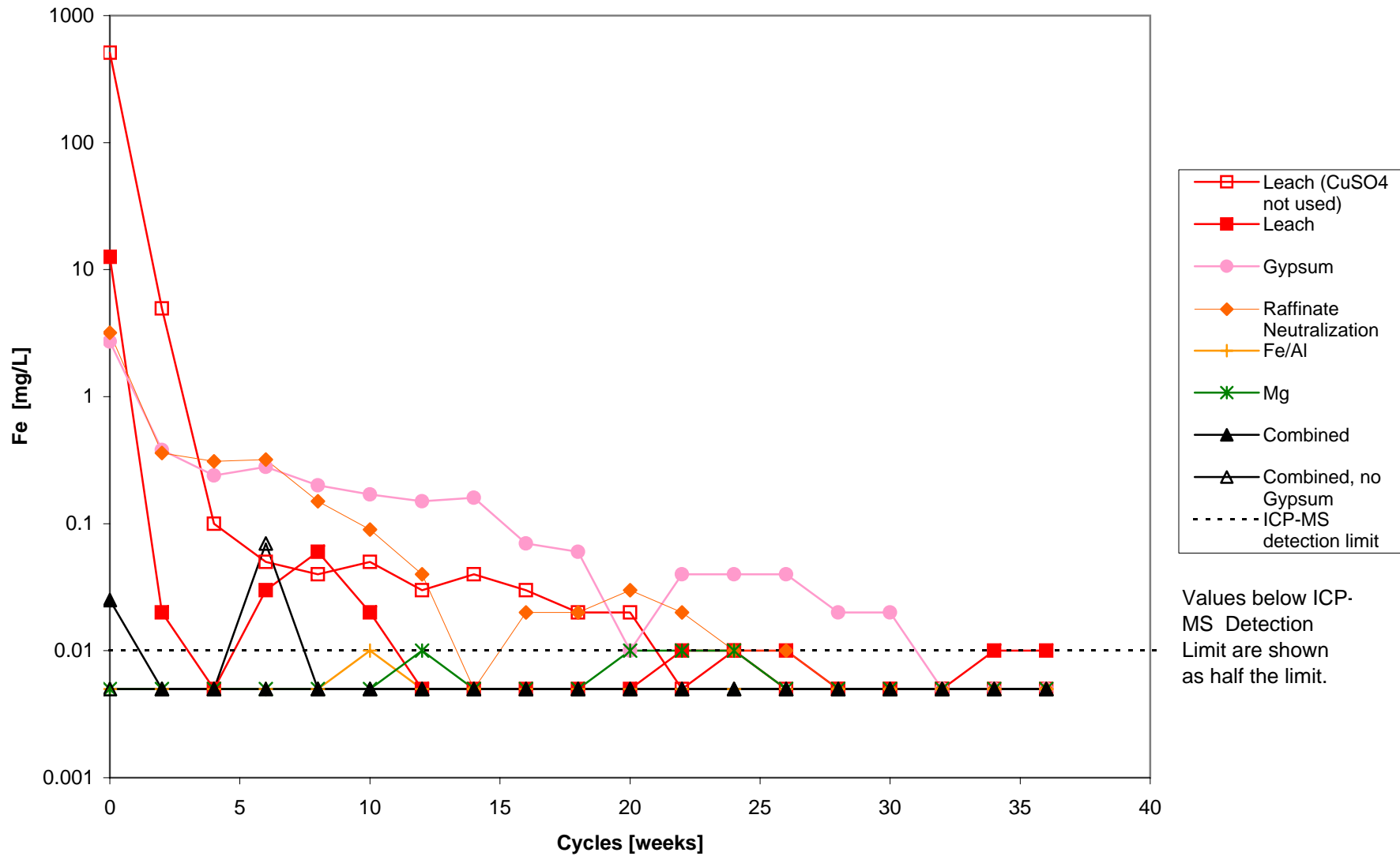


Shake Flasks - Concentrations
NorthMet Project

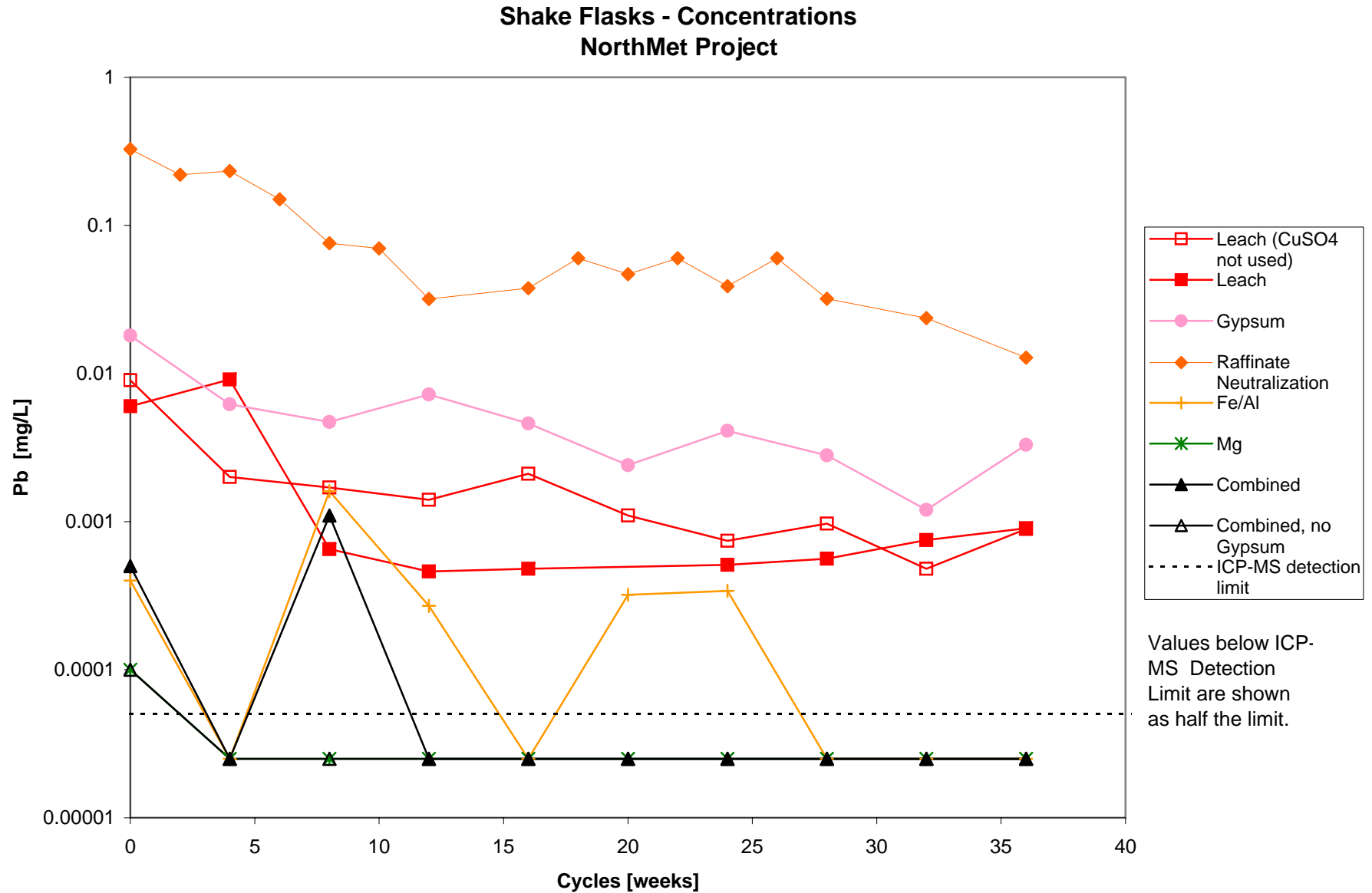


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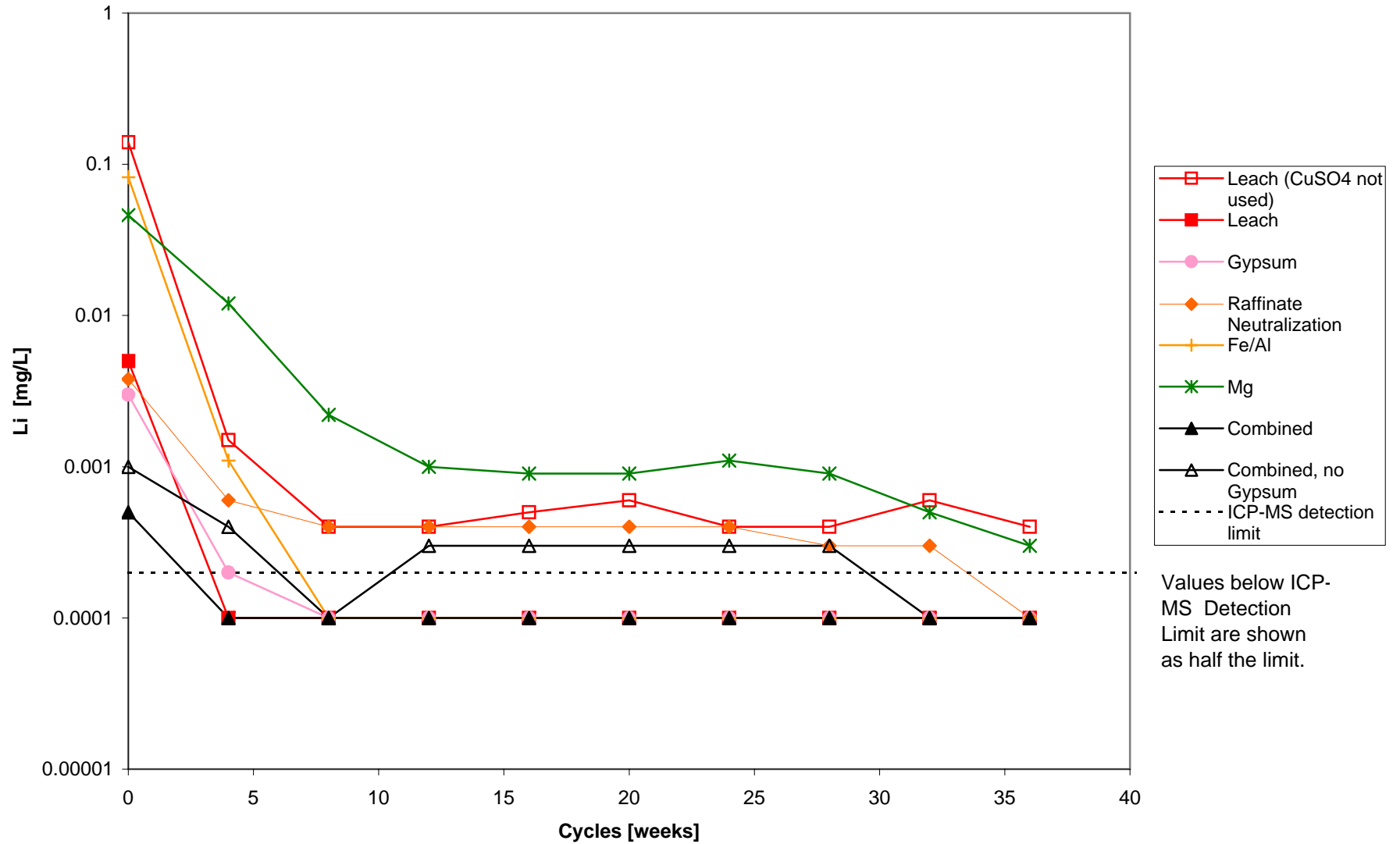
Shake Flasks - Concentrations
NorthMet Project

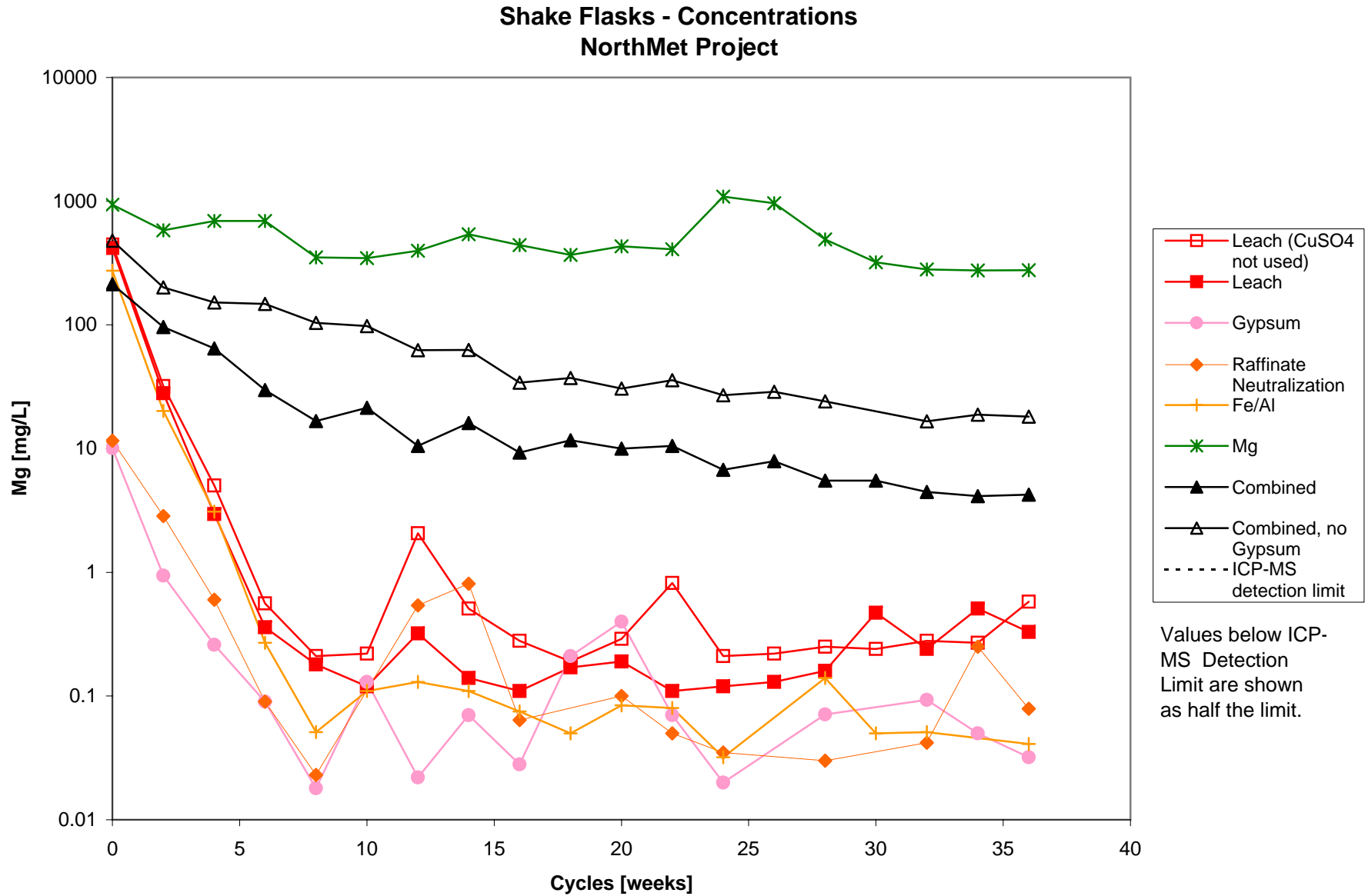


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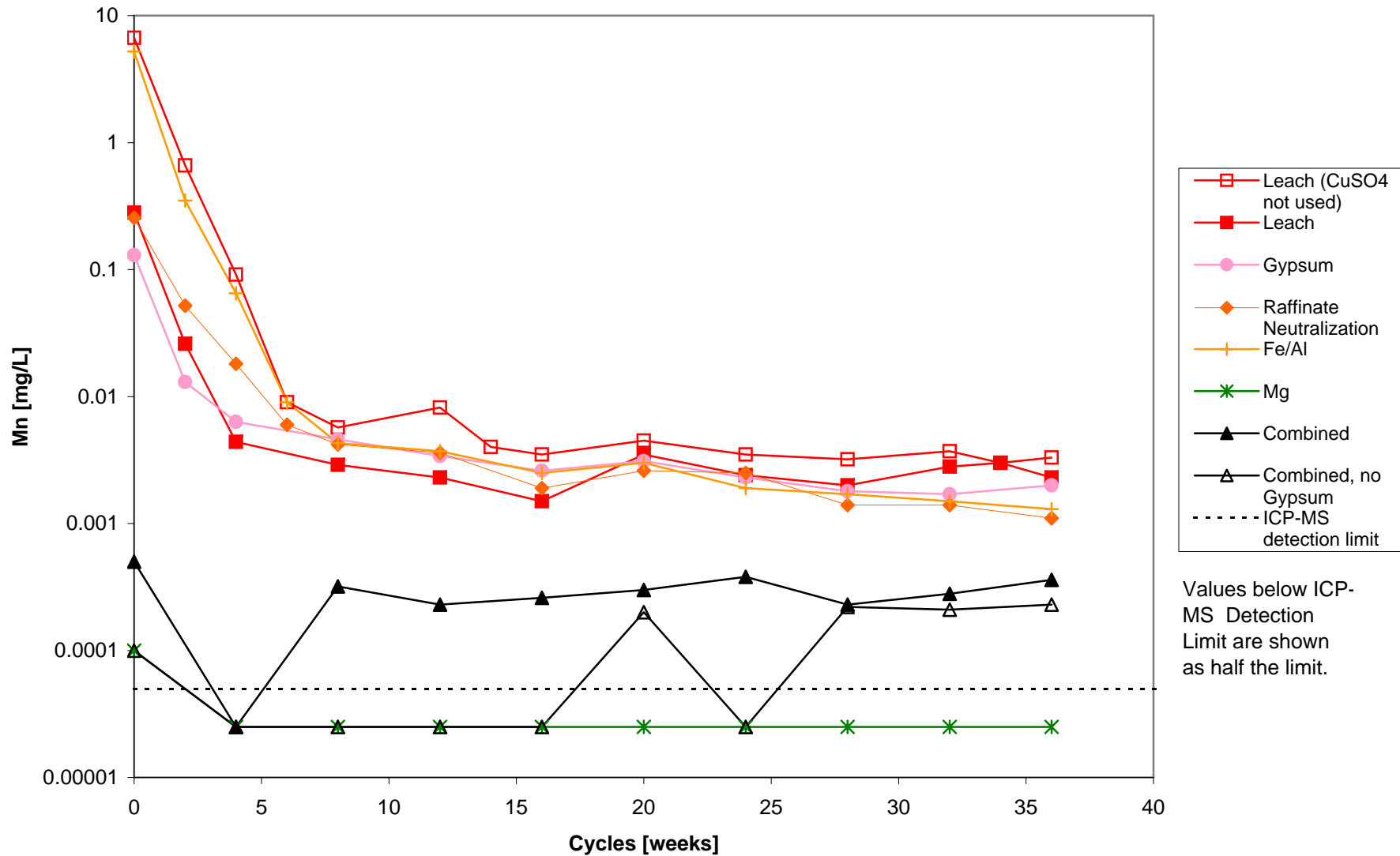


Shake Flasks - Concentrations
NorthMet Project



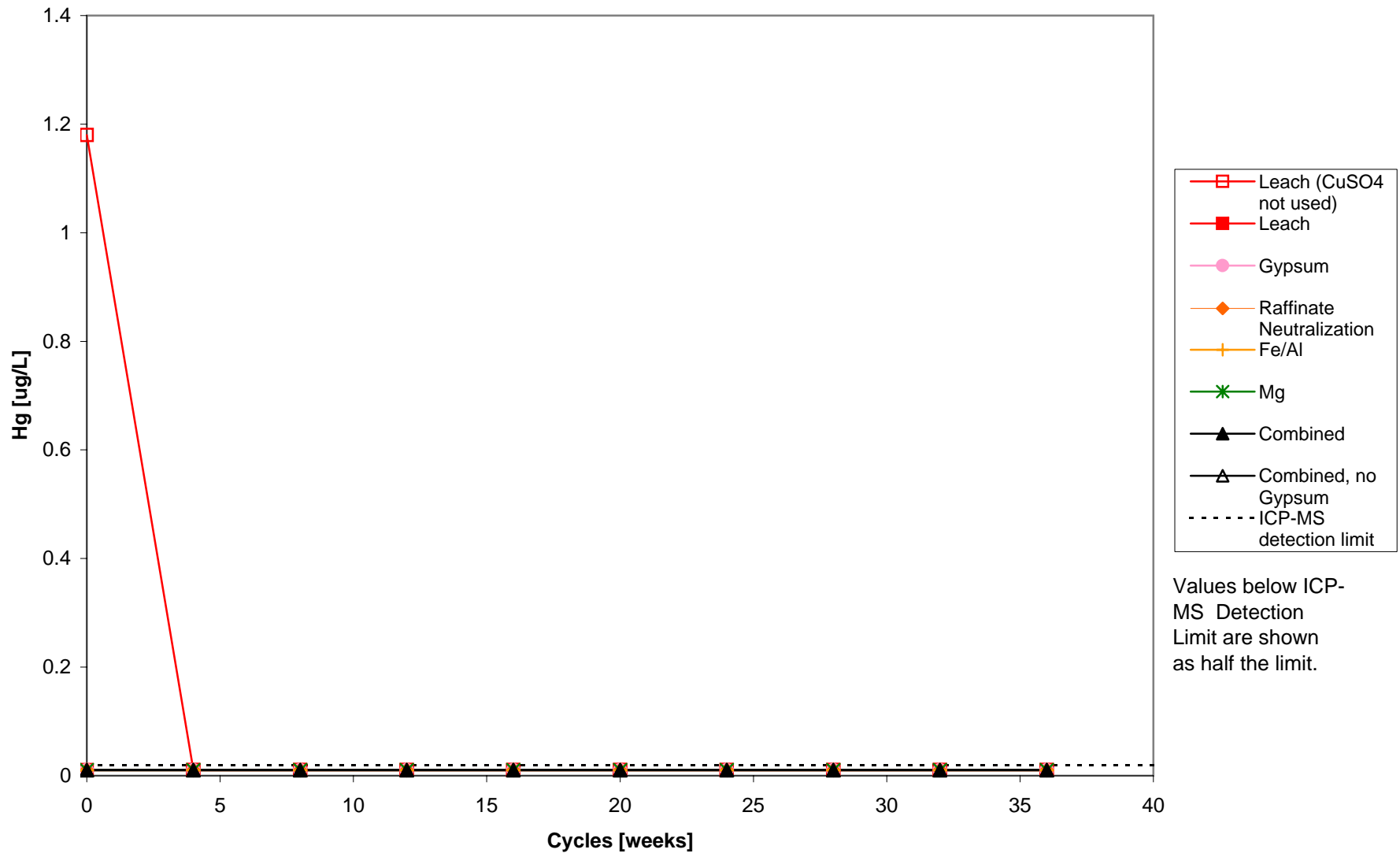


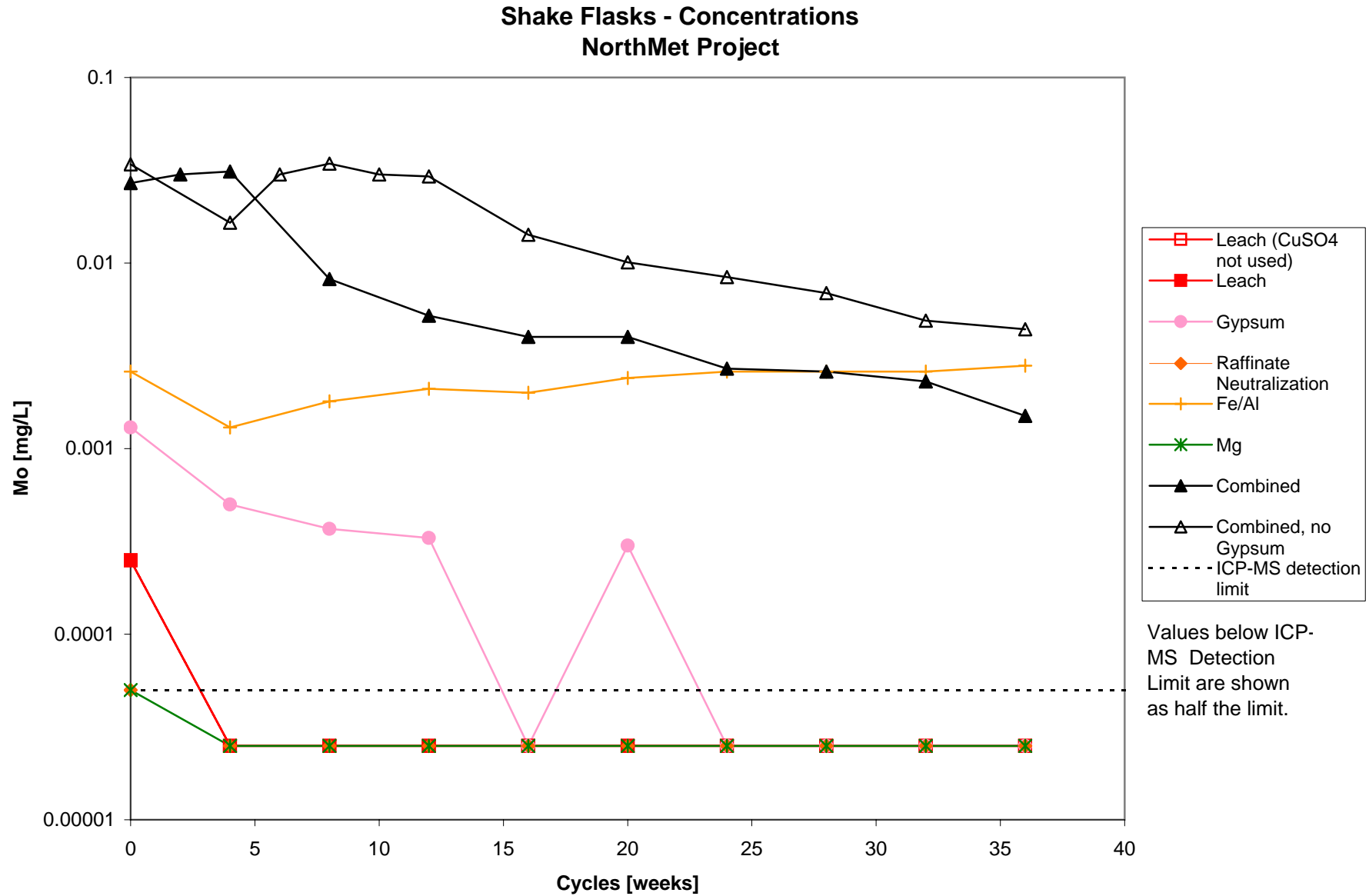
Shake Flasks - Concentrations
NorthMet Project



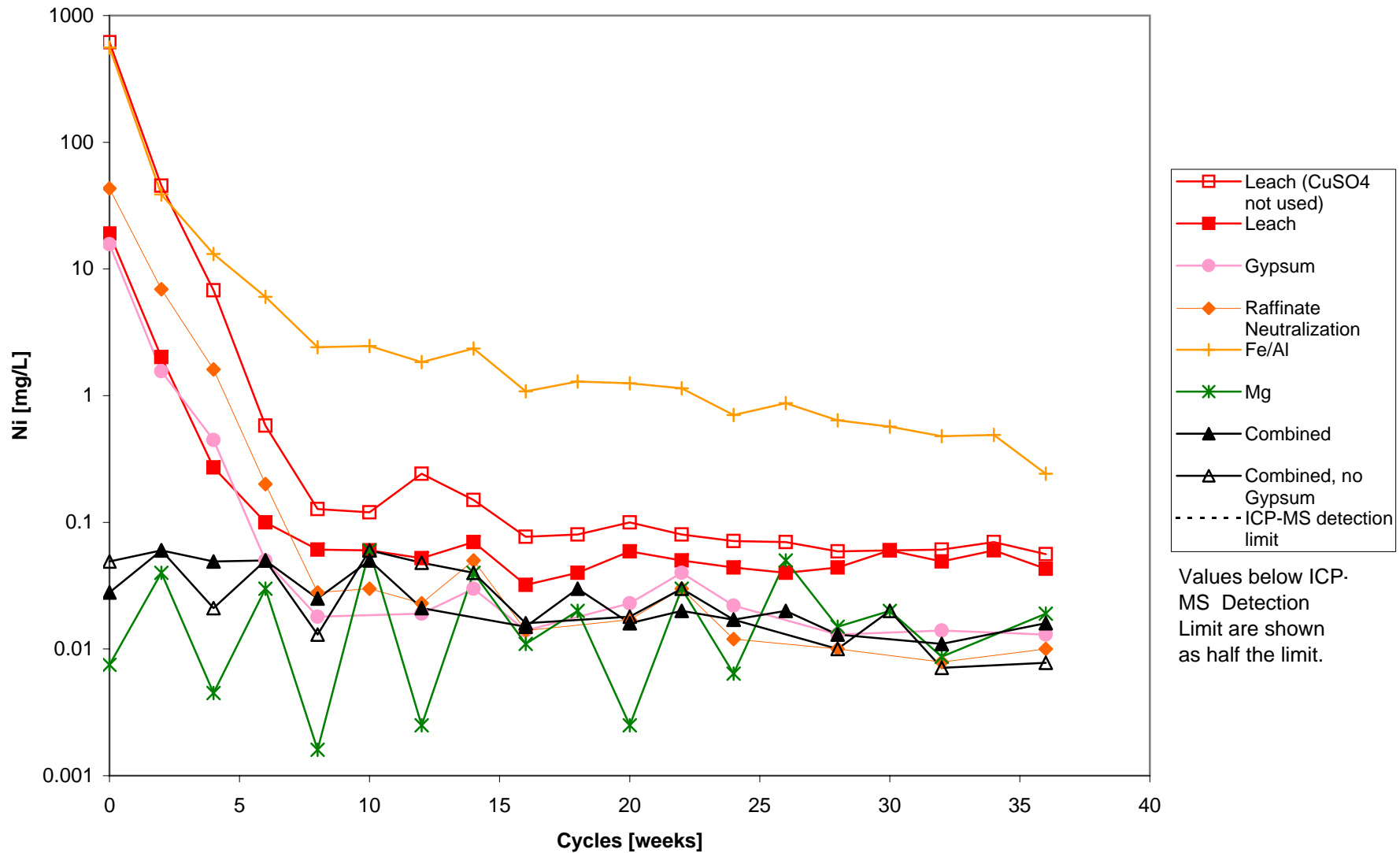
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Shake Flasks - Concentrations
NorthMet Project

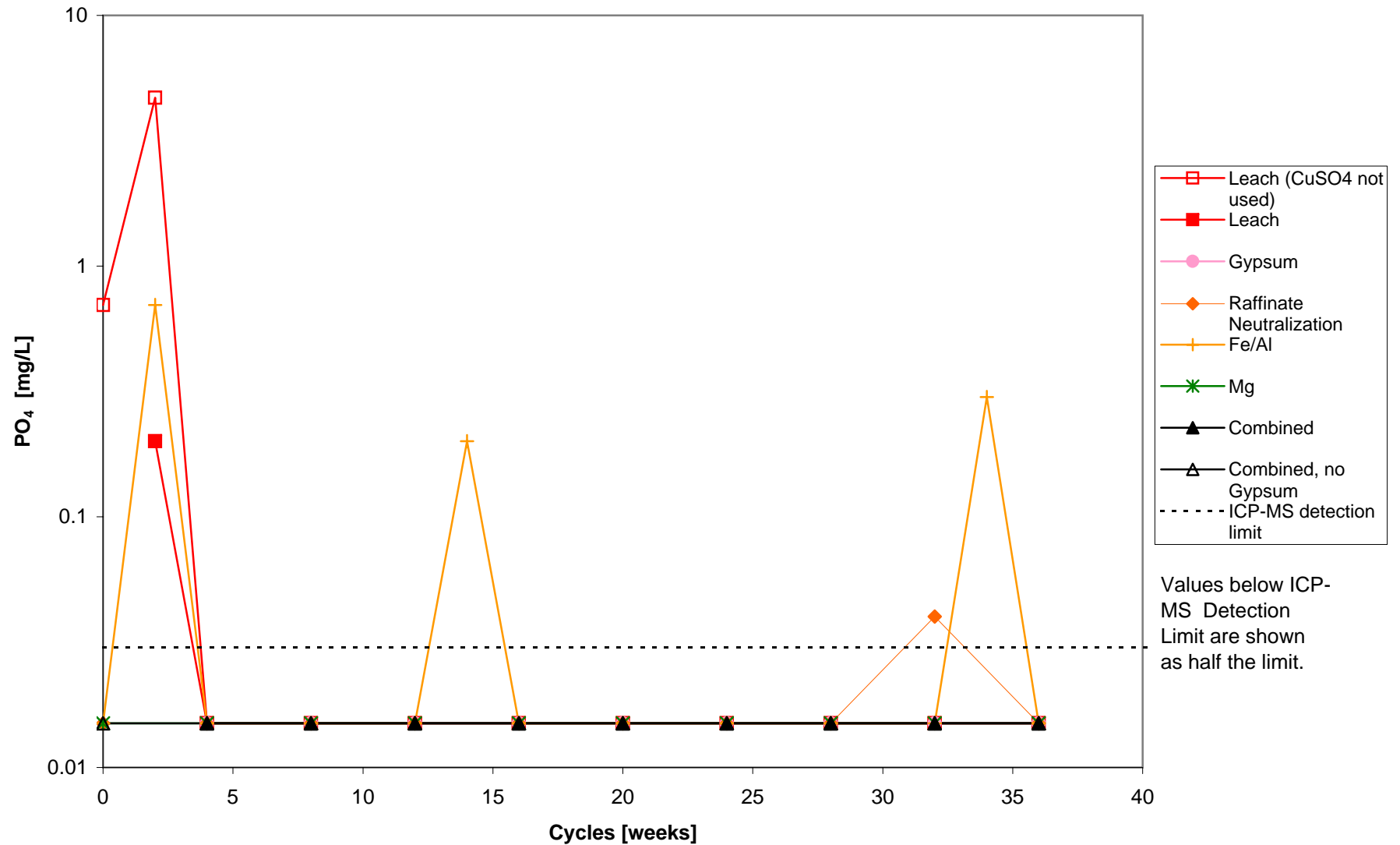




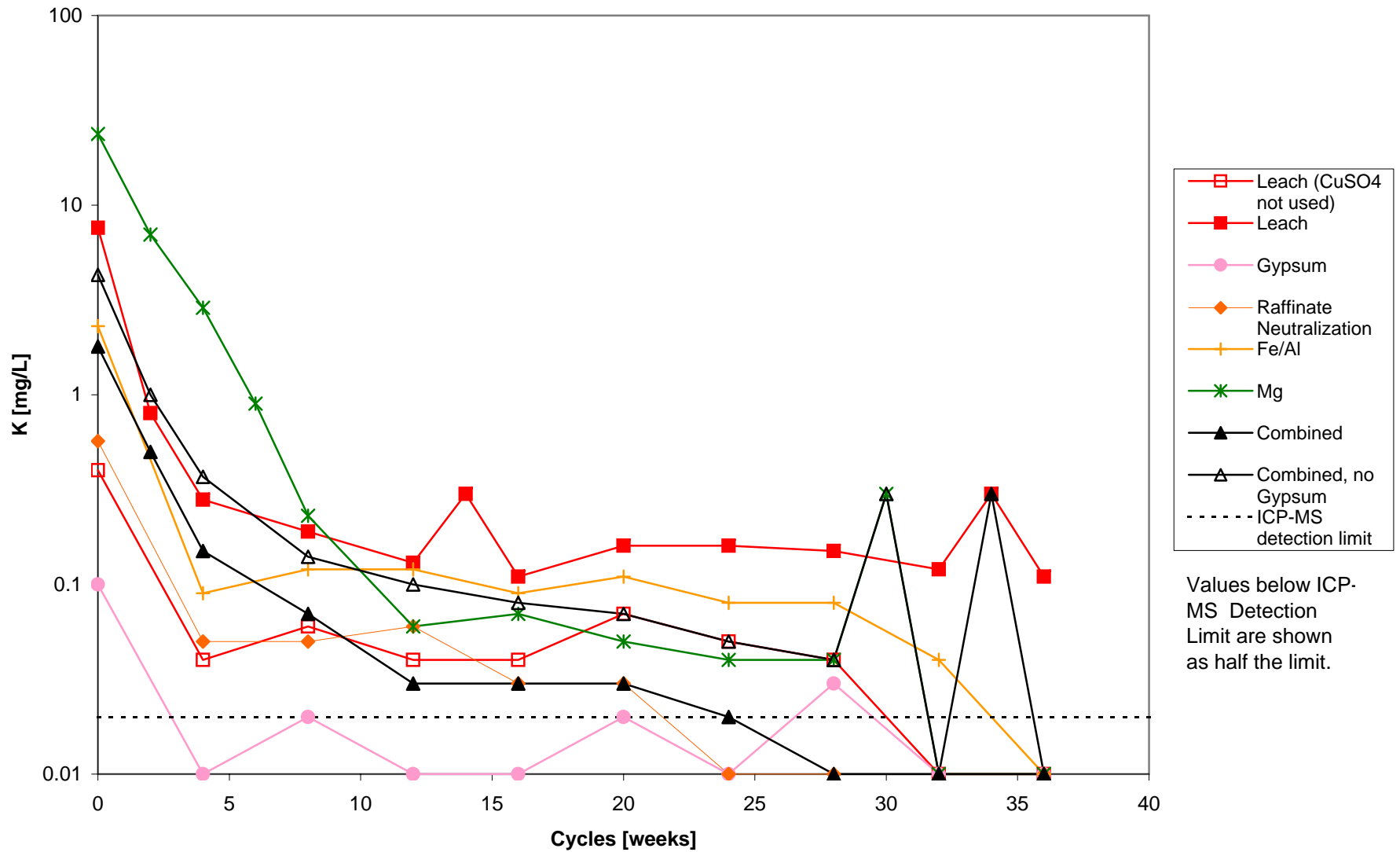
Shake Flasks - Concentrations
NorthMet Project

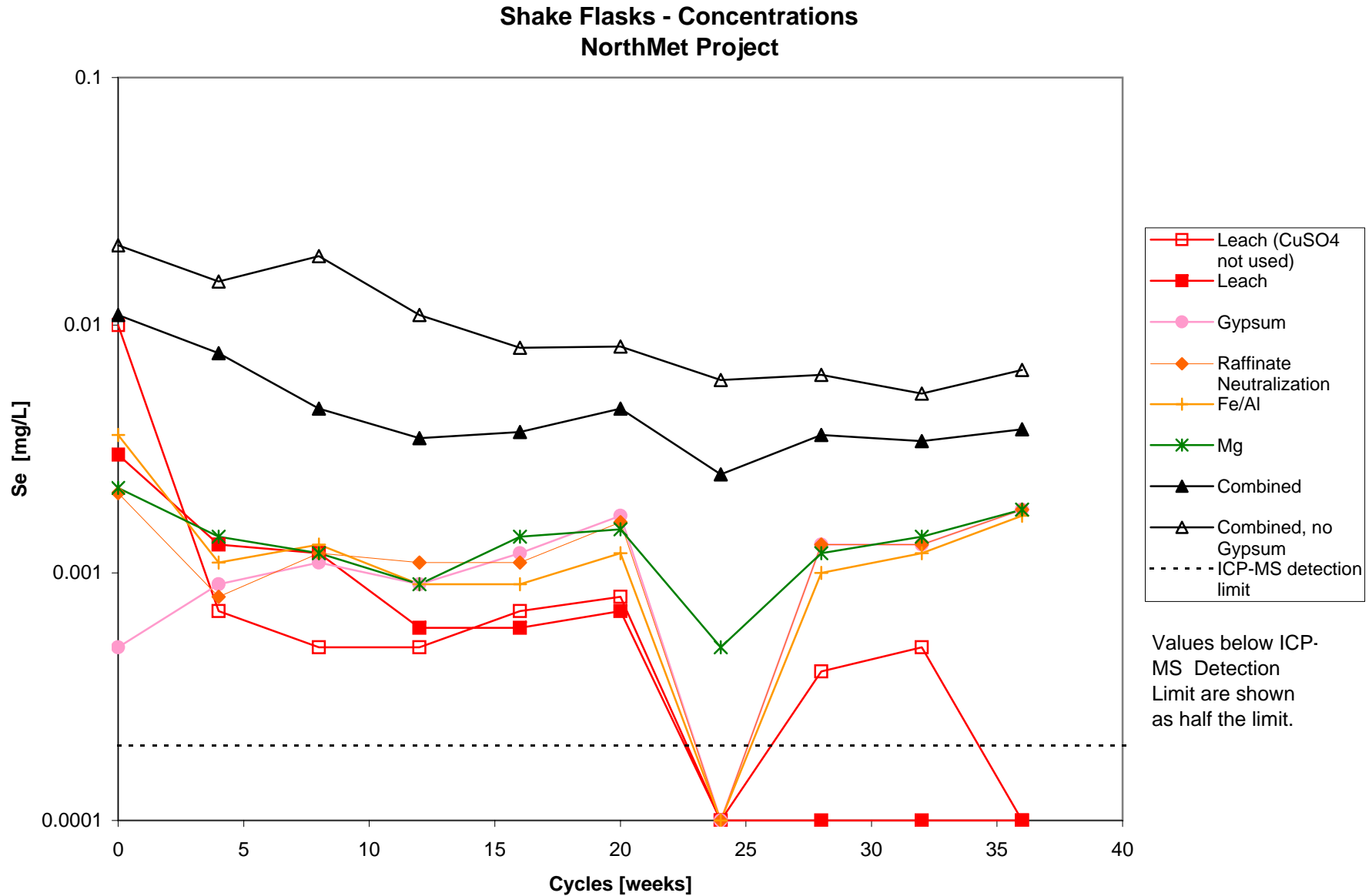


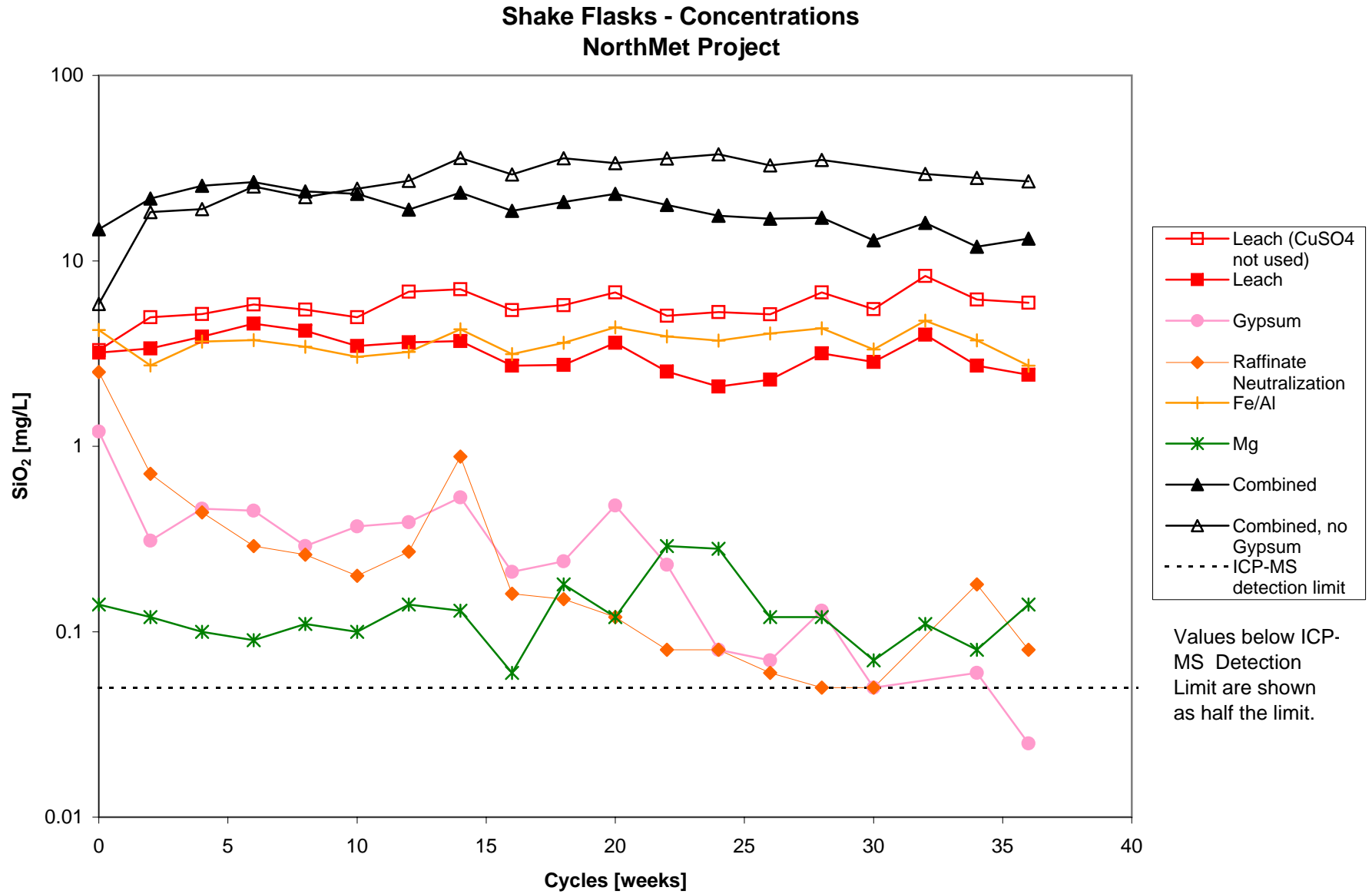
Shake Flasks - Concentrations
NorthMet Project



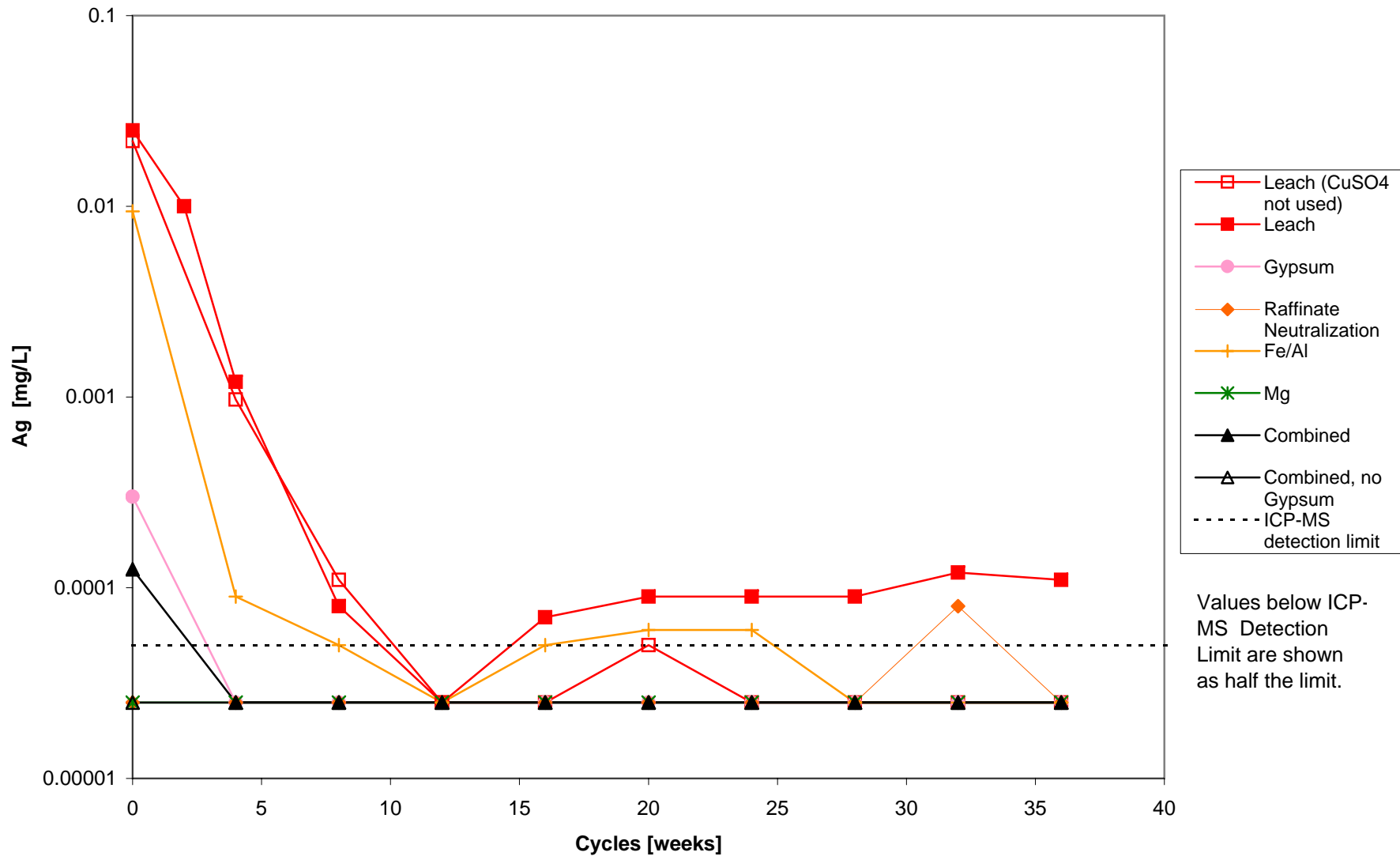
Shake Flasks - Concentrations
NorthMet Project





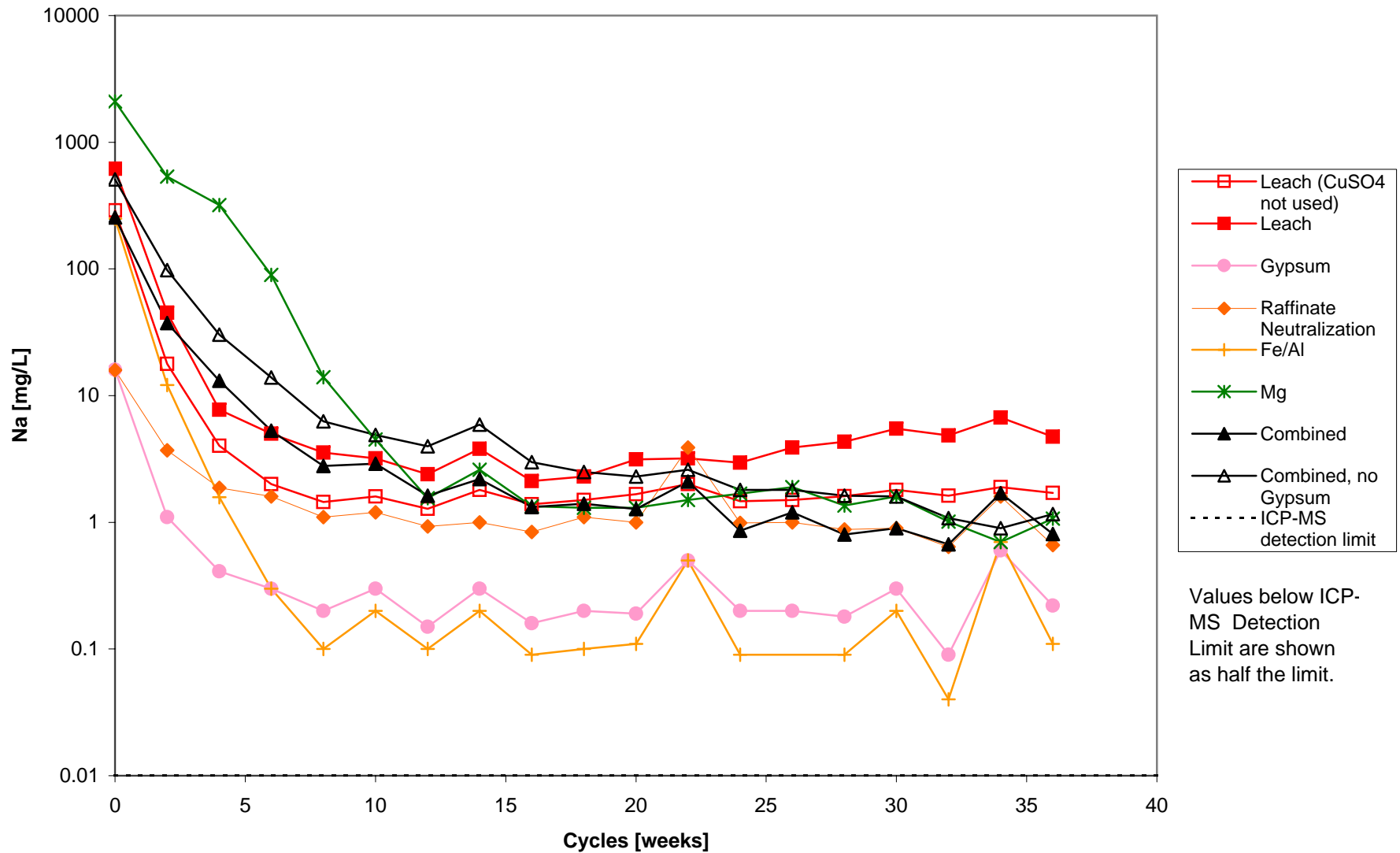


Shake Flasks - Concentrations
NorthMet Project



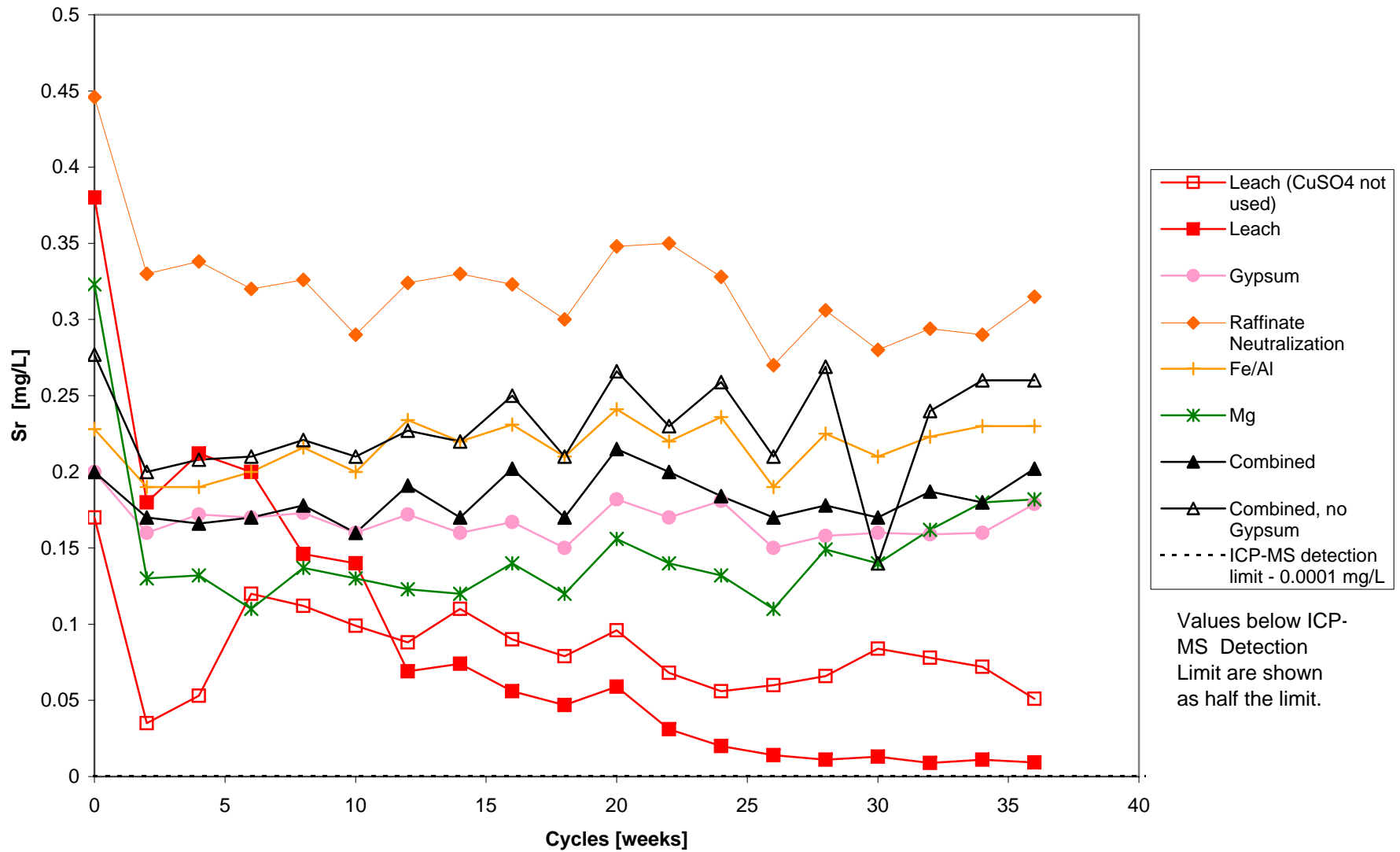
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Shake Flasks - Concentrations
NorthMet Project

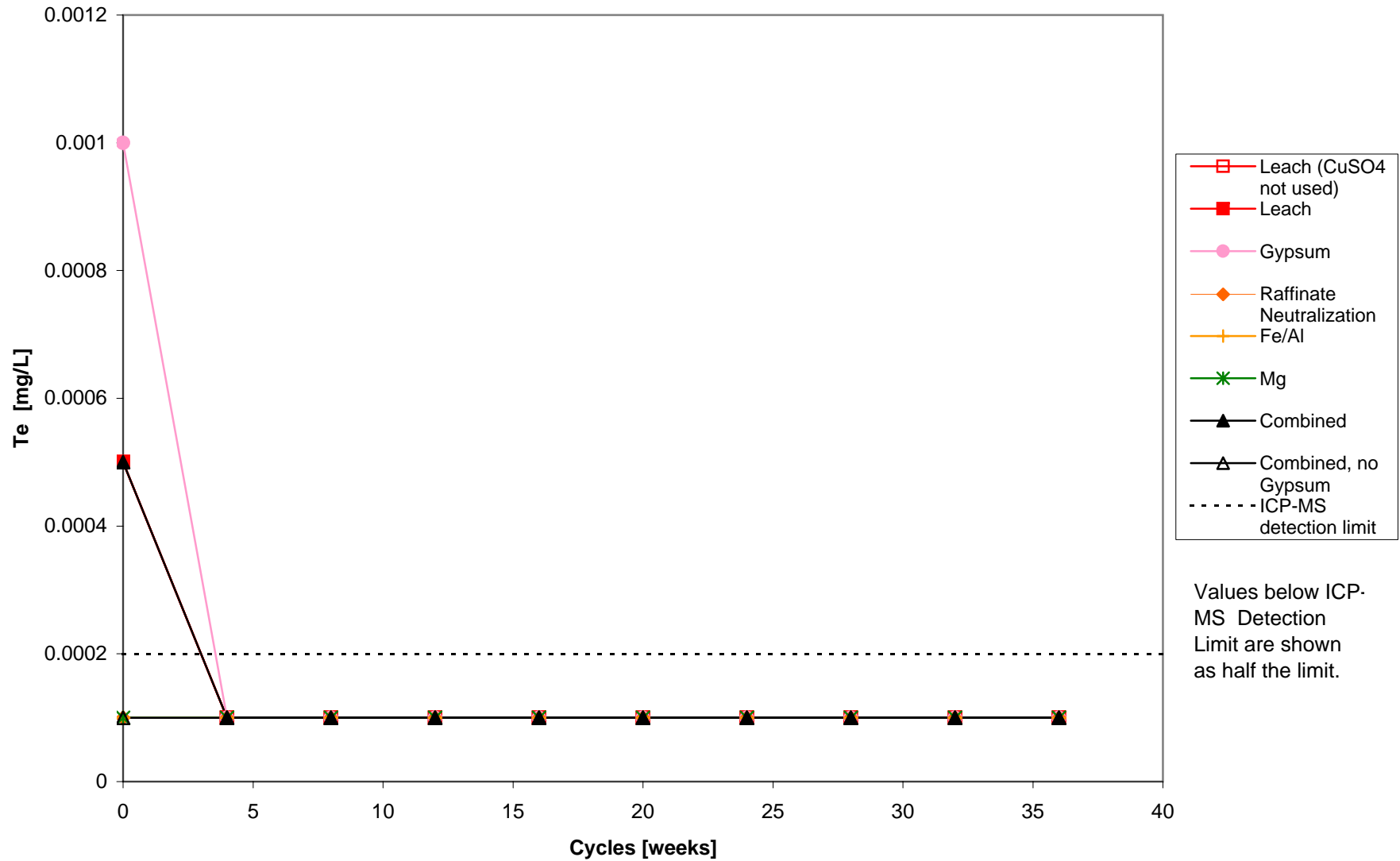


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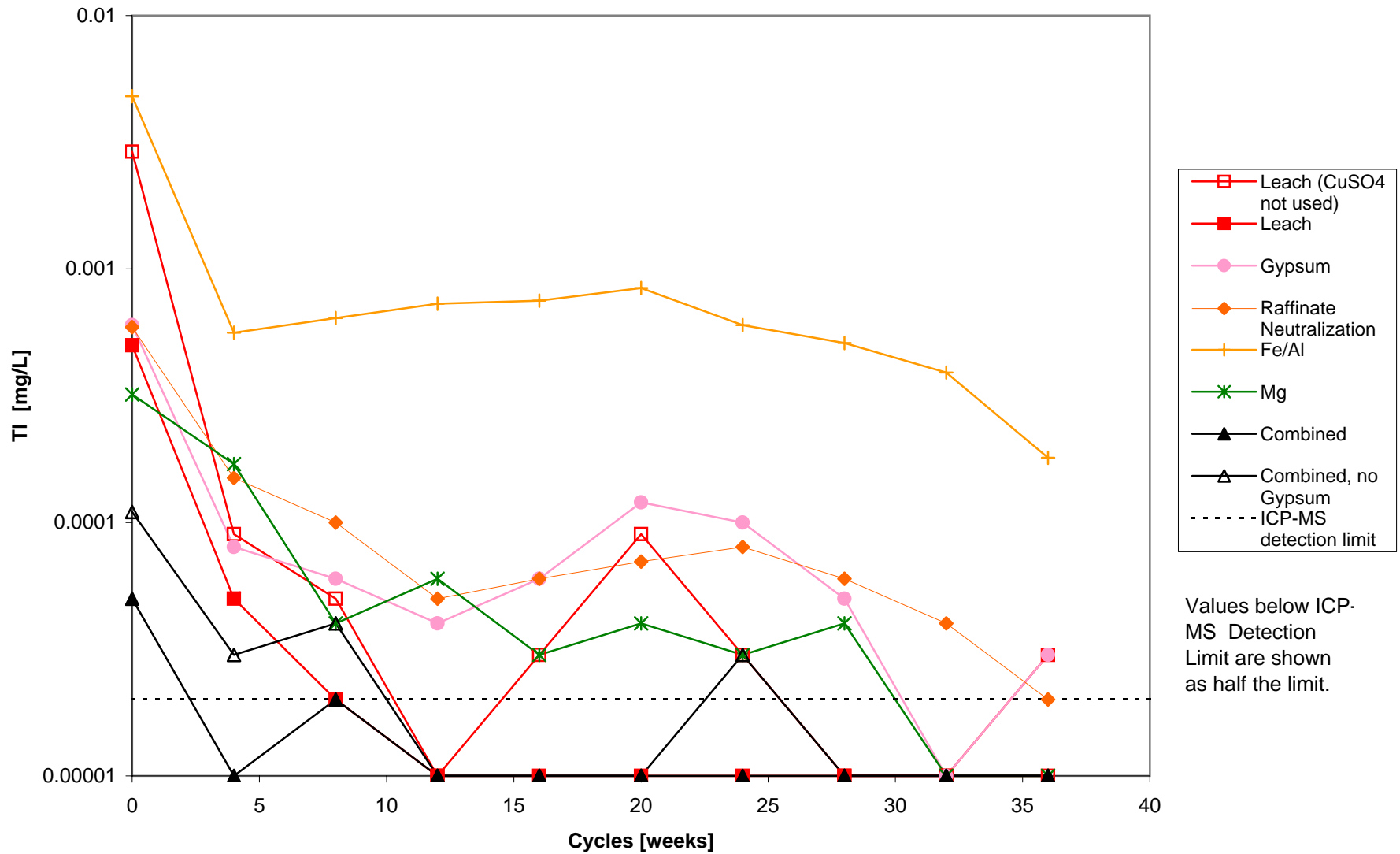
Shake Flasks - Concentrations
NorthMet Project



Shake Flasks - Concentrations
NorthMet Project

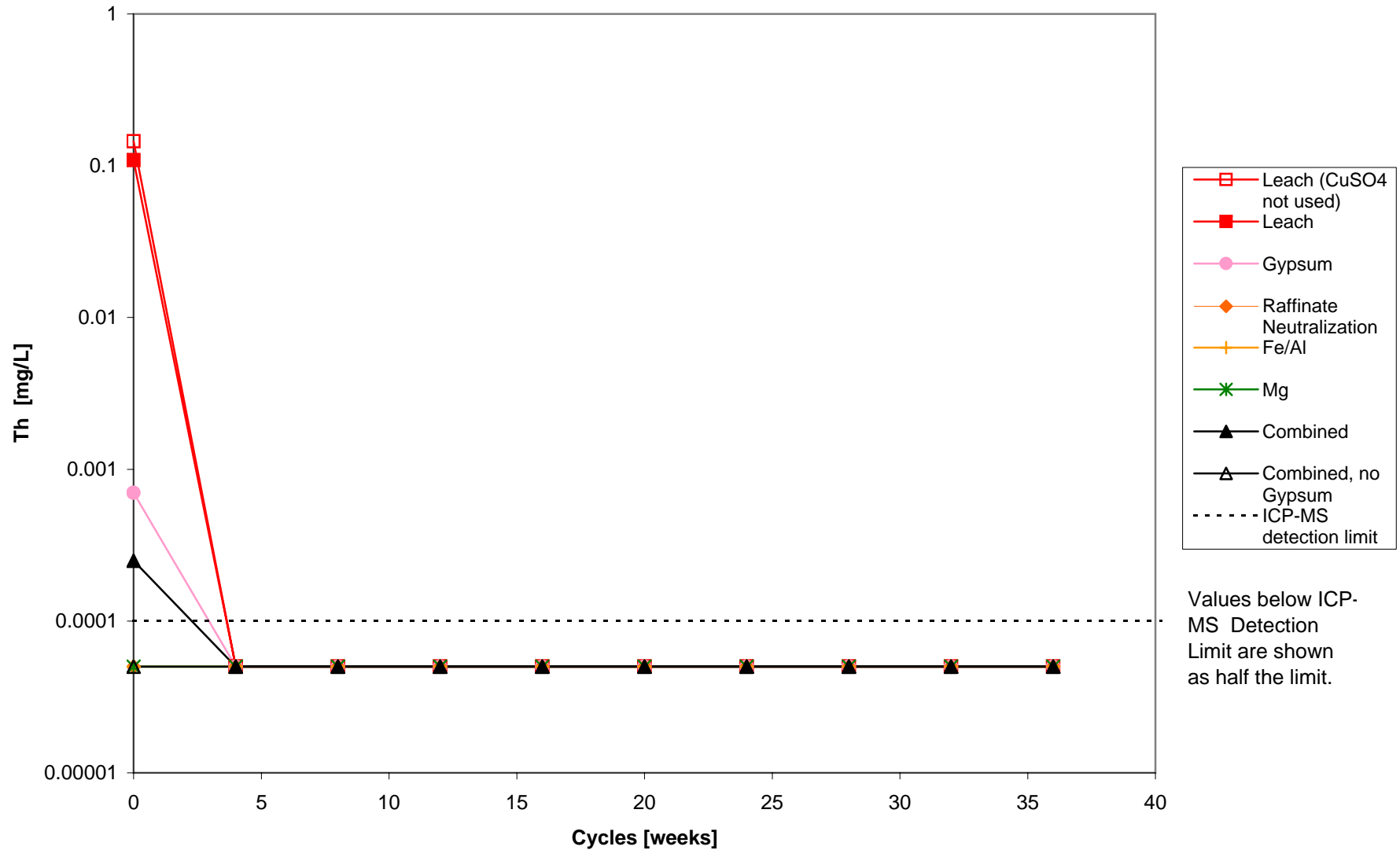


Shake Flasks - Concentrations
NorthMet Project

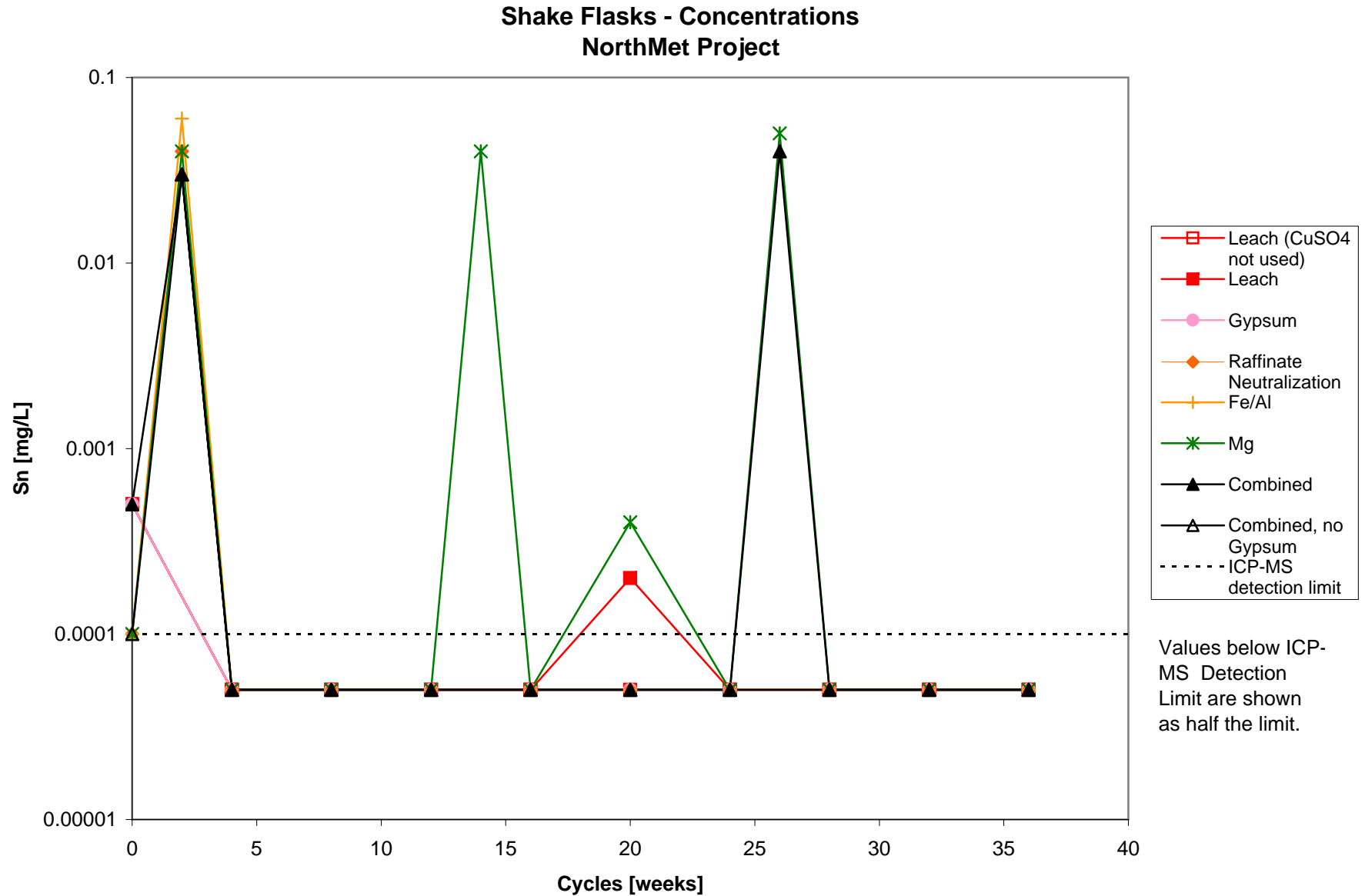


Values below ICP-MS Detection Limit are shown as half the limit.

Shake Flasks - Concentrations
NorthMet Project

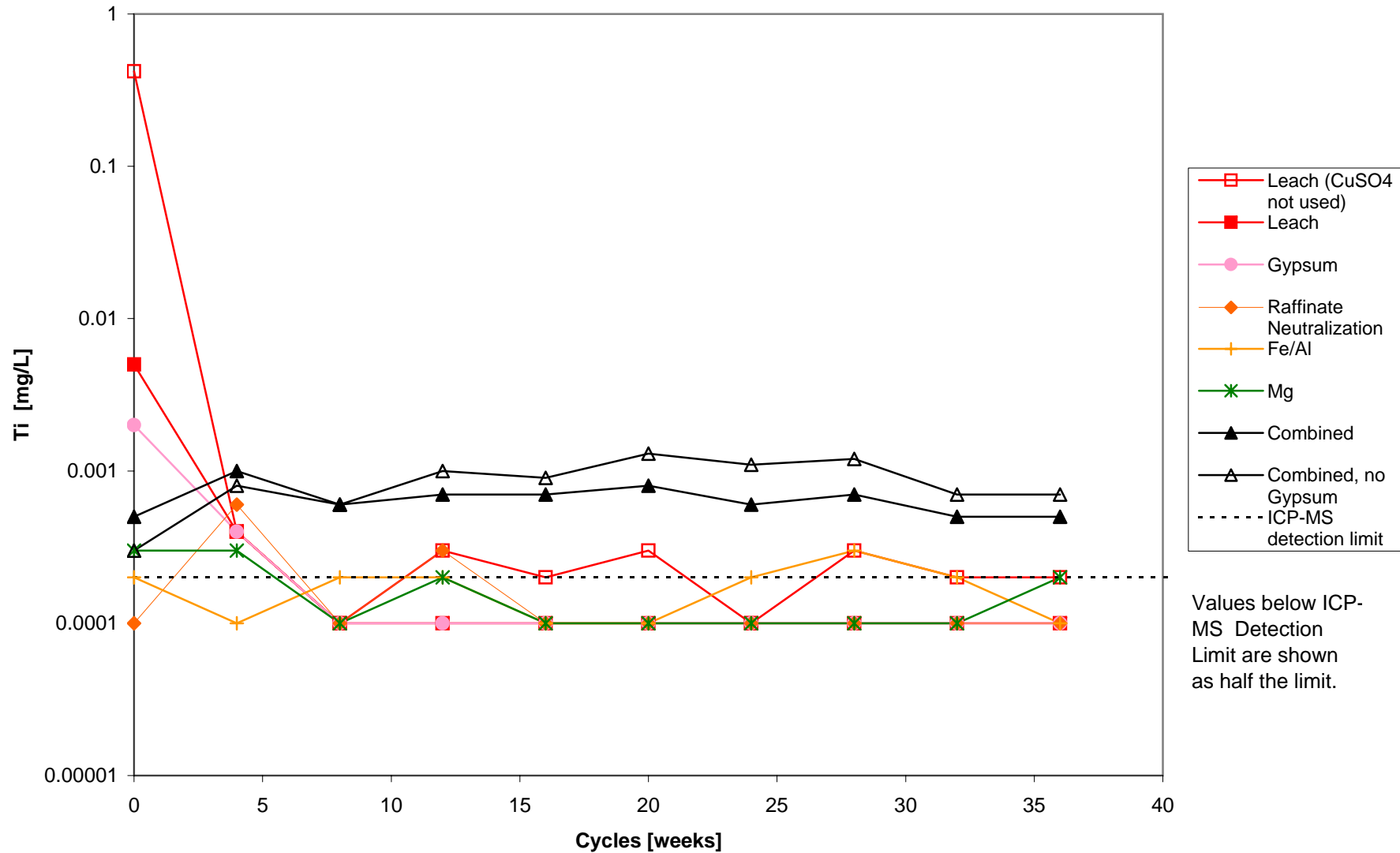


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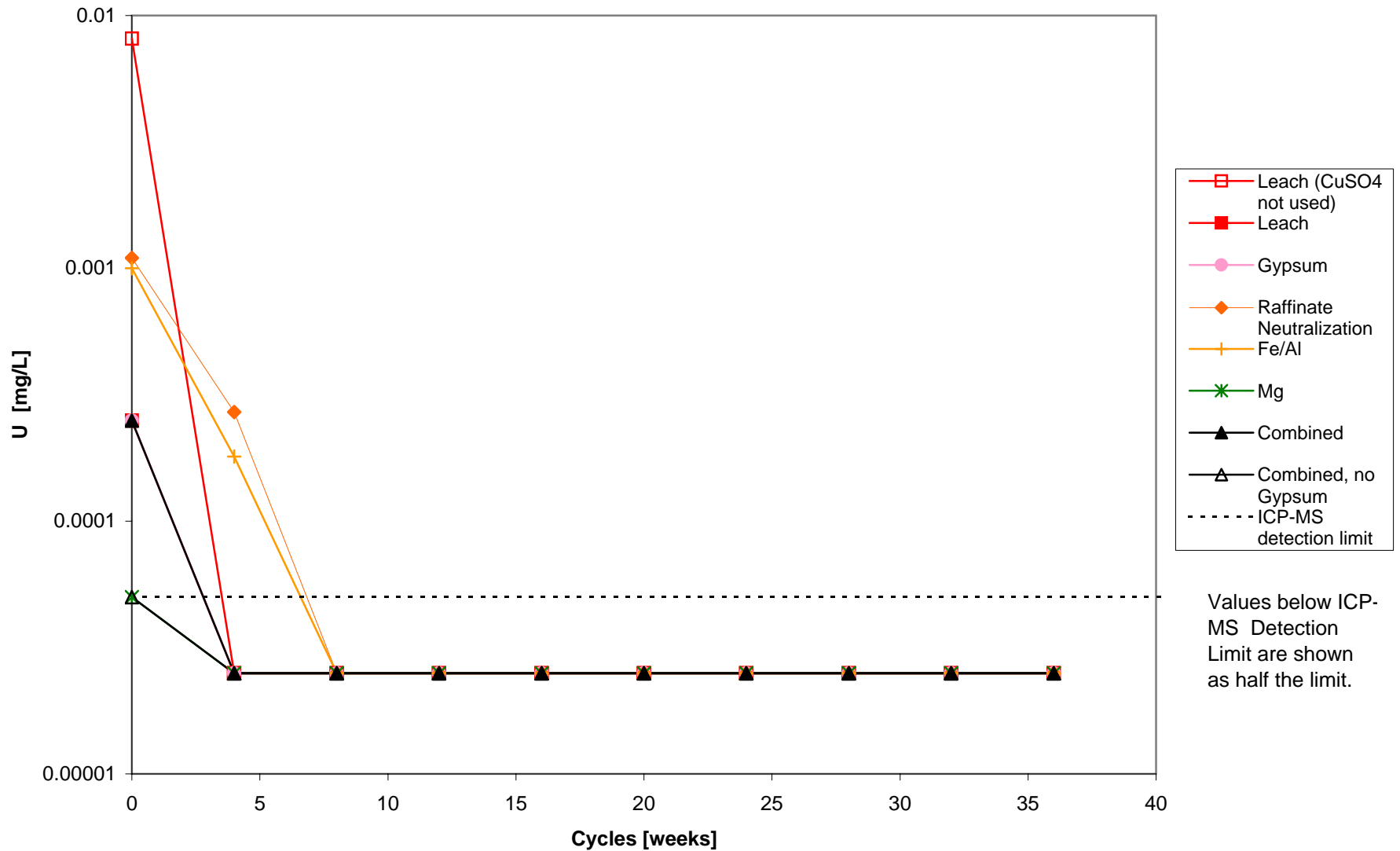
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Shake Flasks - Concentrations
NorthMet Project



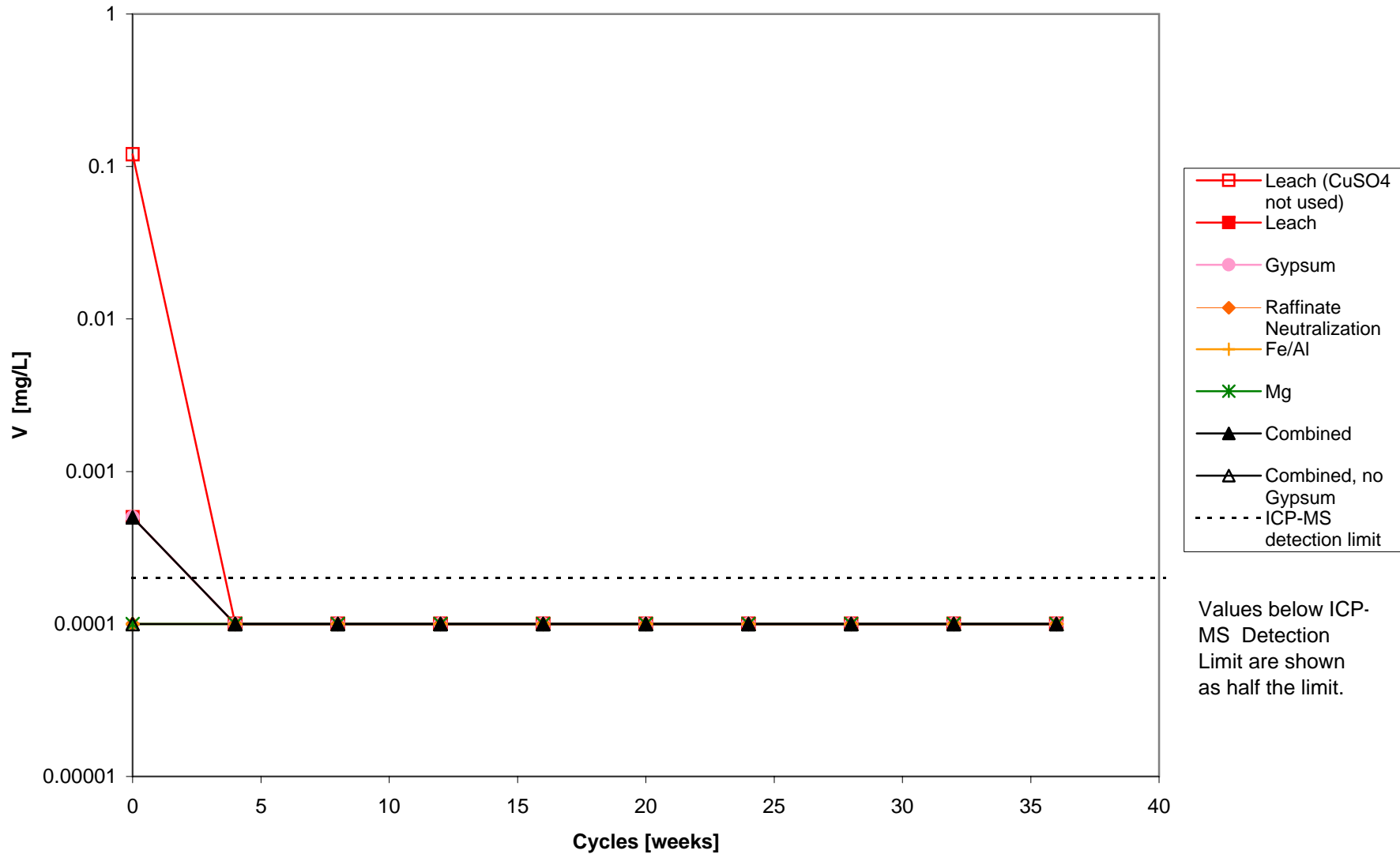
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Shake Flasks - Concentrations
NorthMet Project

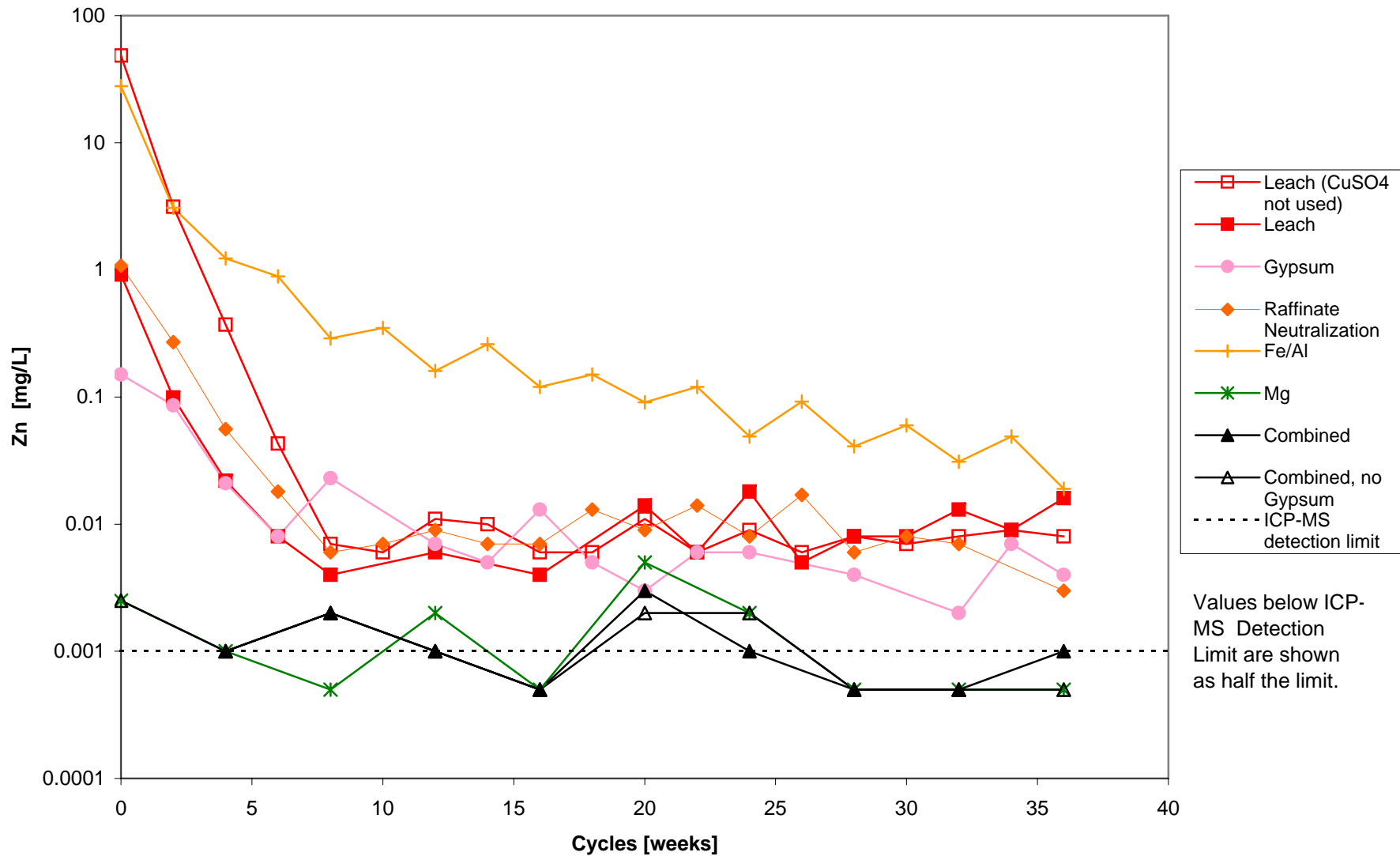


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Shake Flasks - Concentrations
NorthMet Project

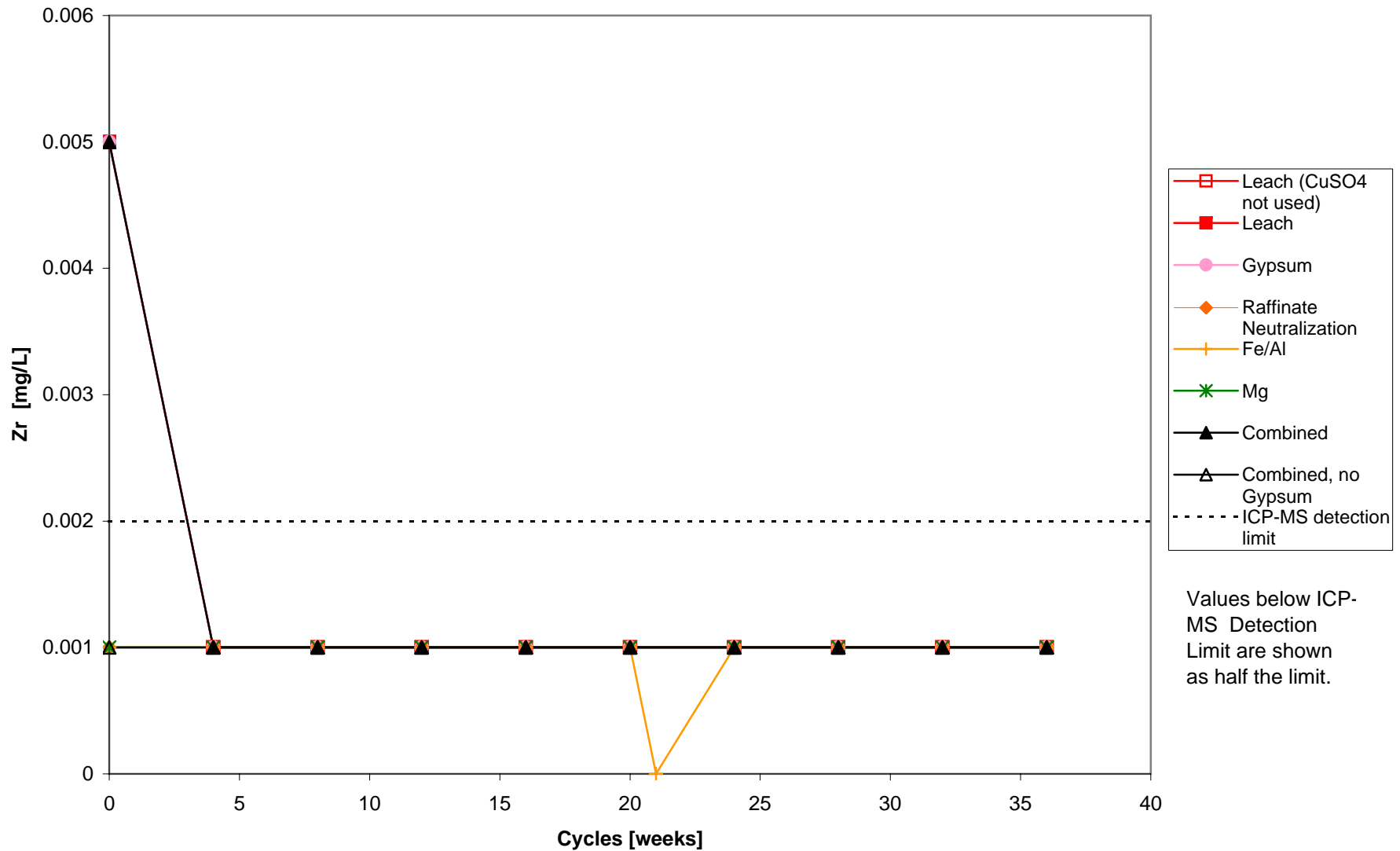


Shake Flasks - Concentrations
NorthMet Project



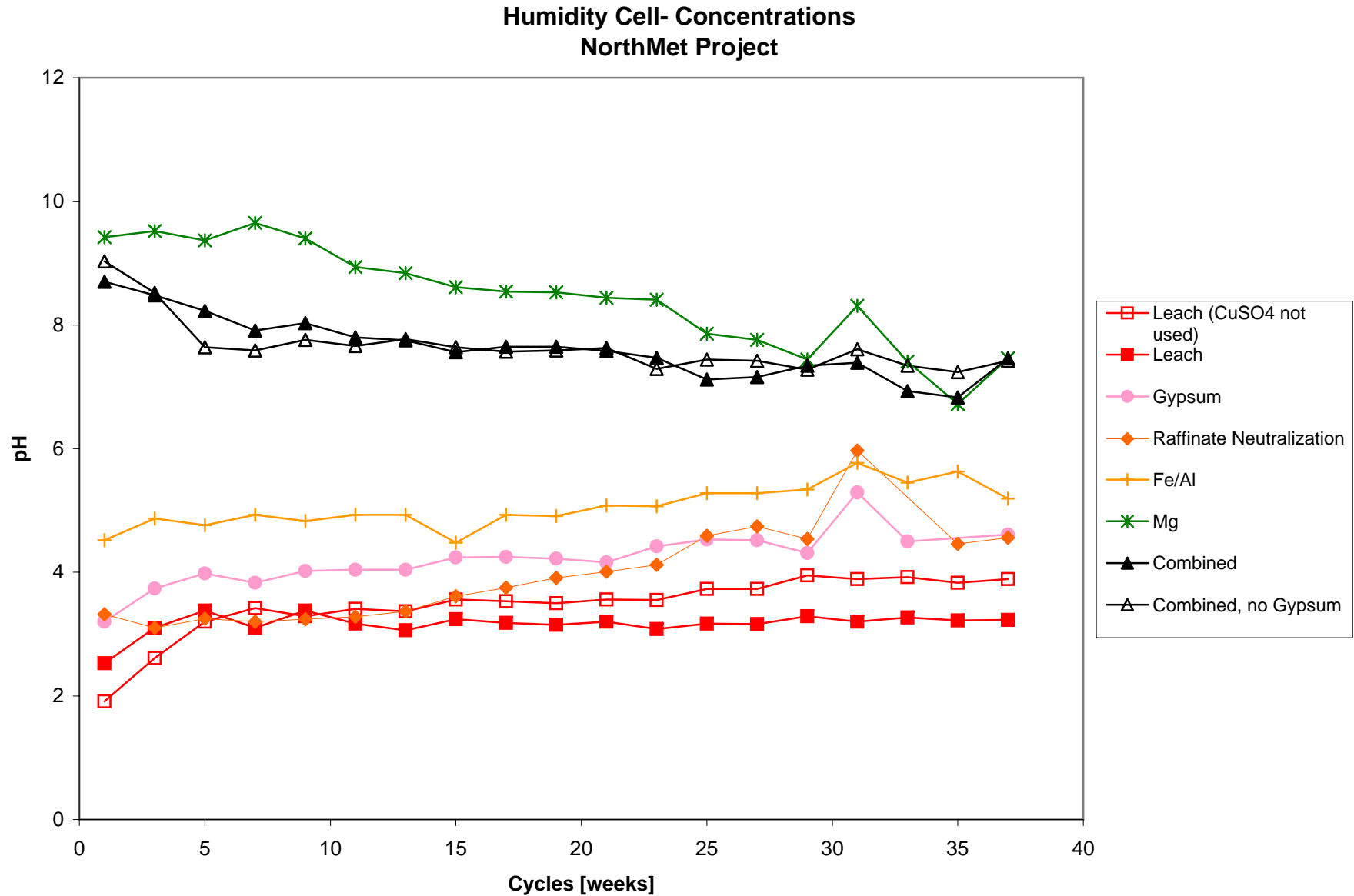
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Shake Flasks - Concentrations
NorthMet Project

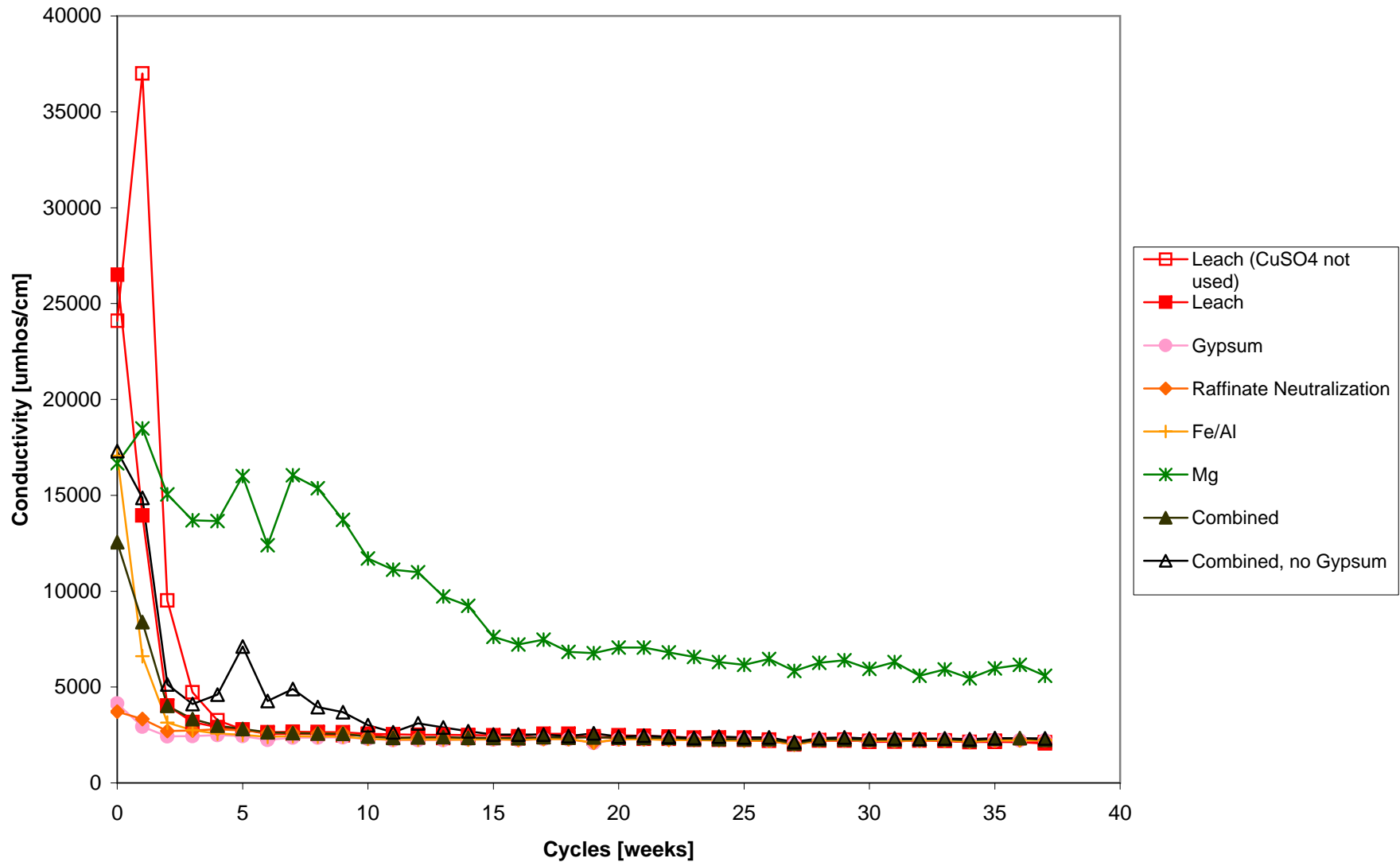


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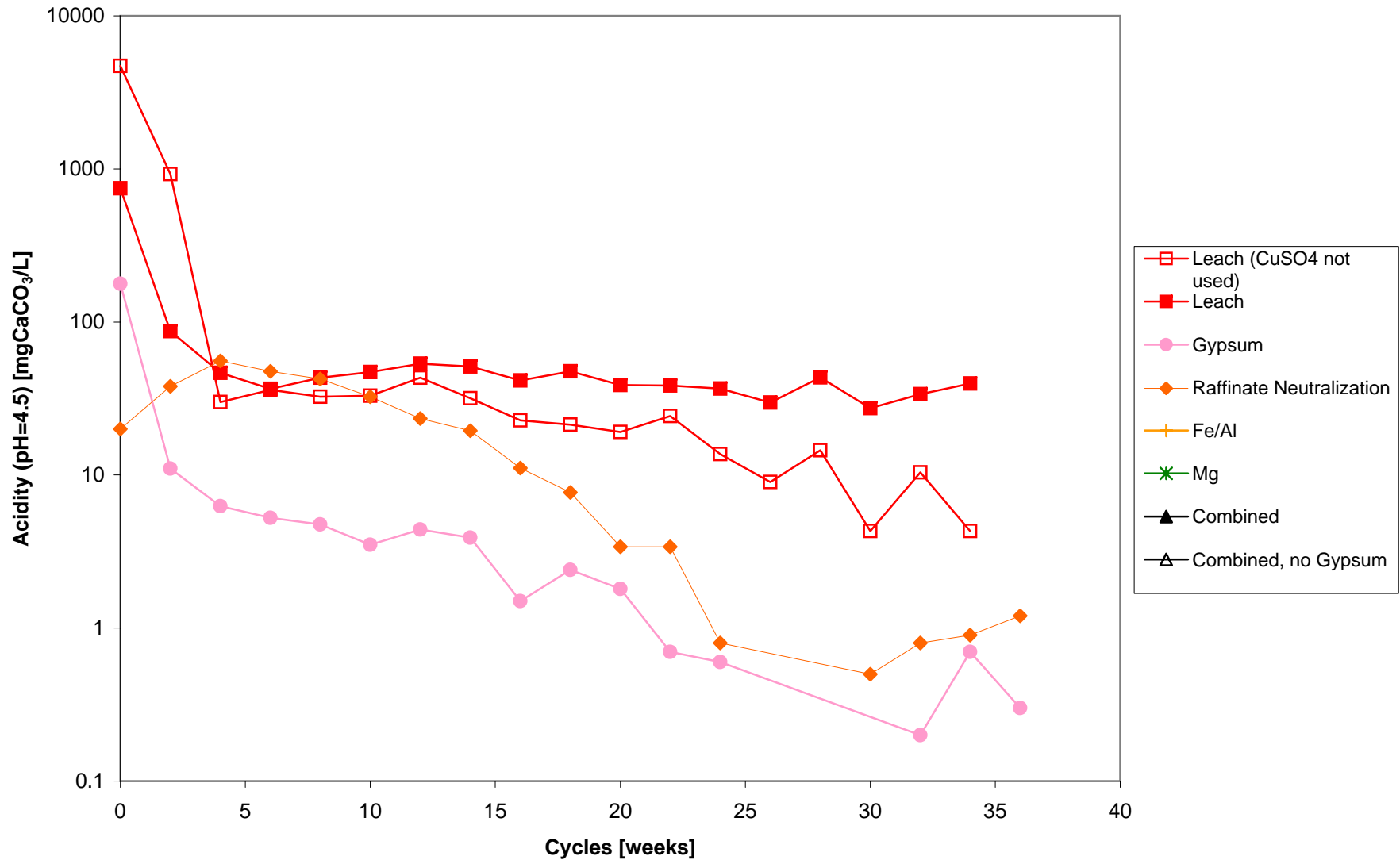
Appendix C.2
Humidity Cells



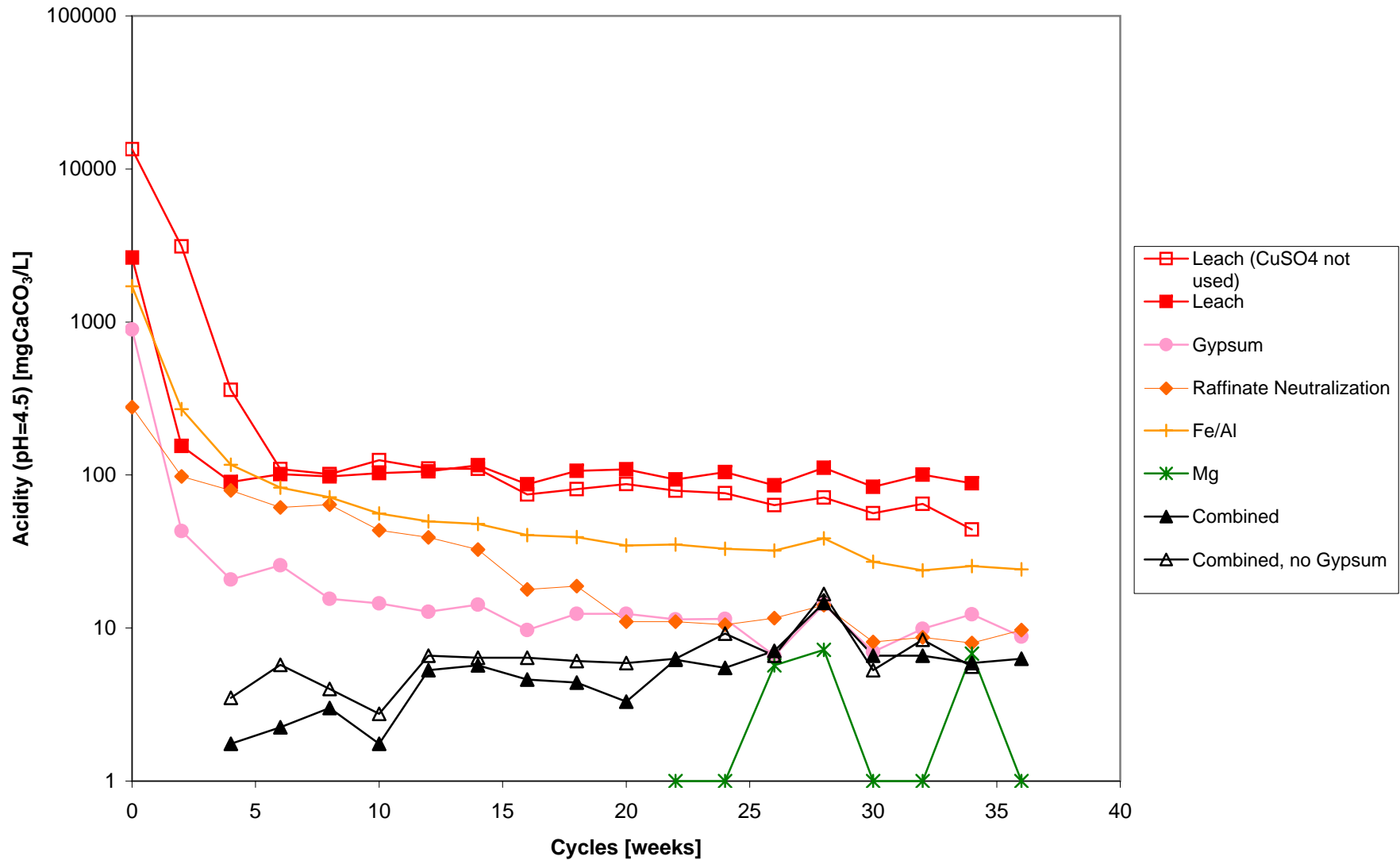
Humidity Cell - Concentrations
NorthMet Project



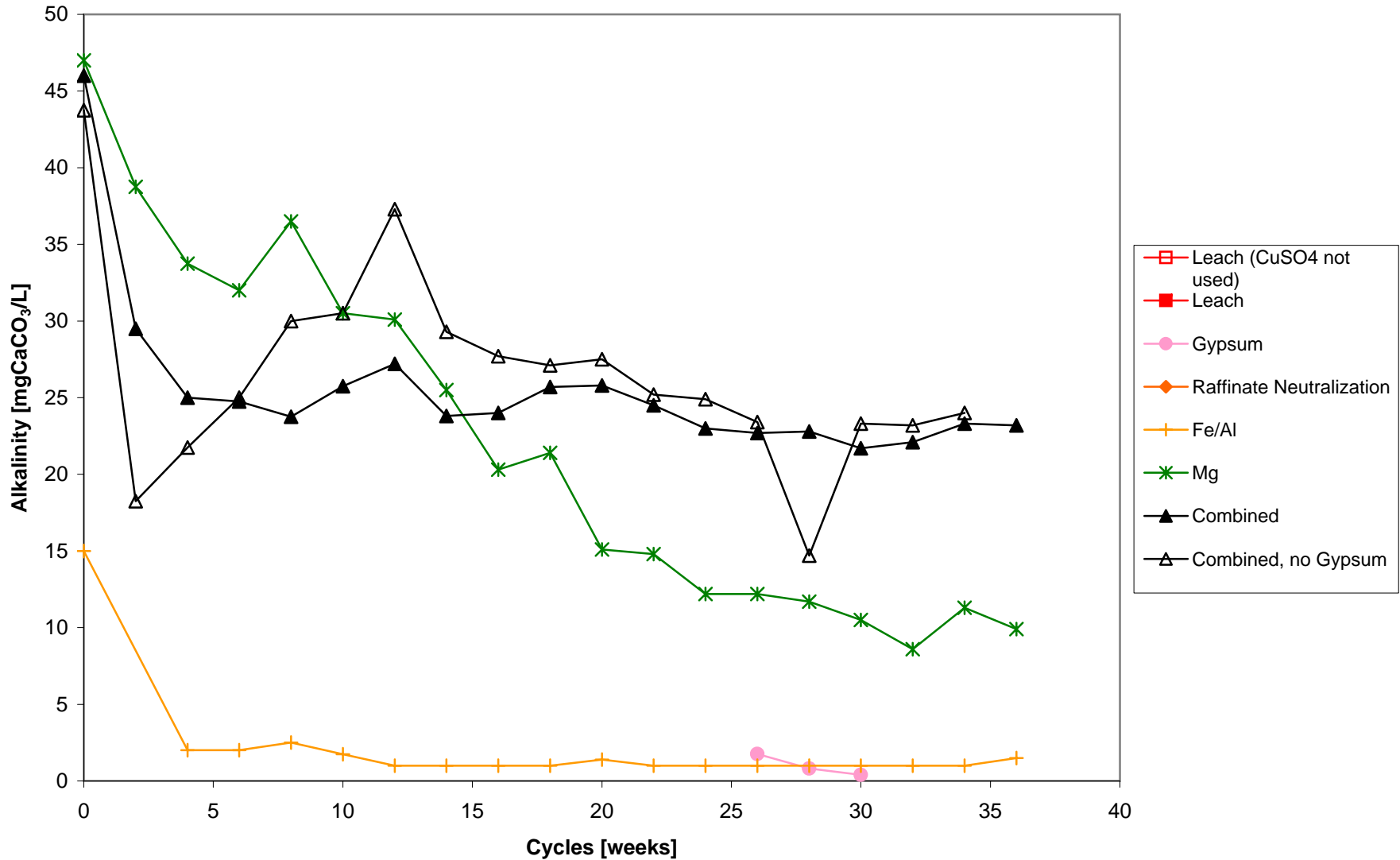
Humidity Cell - Concentrations
NorthMet Project

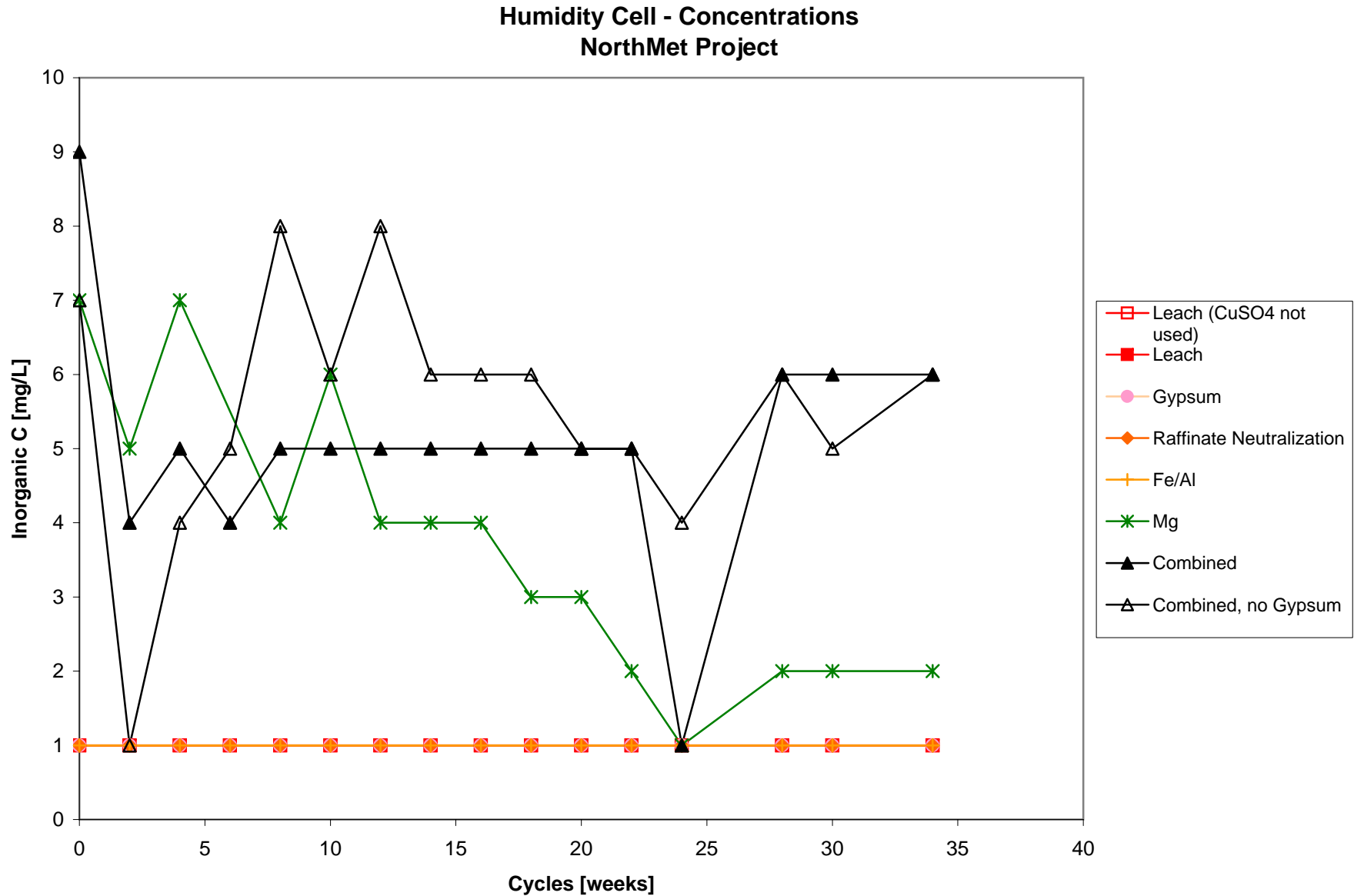


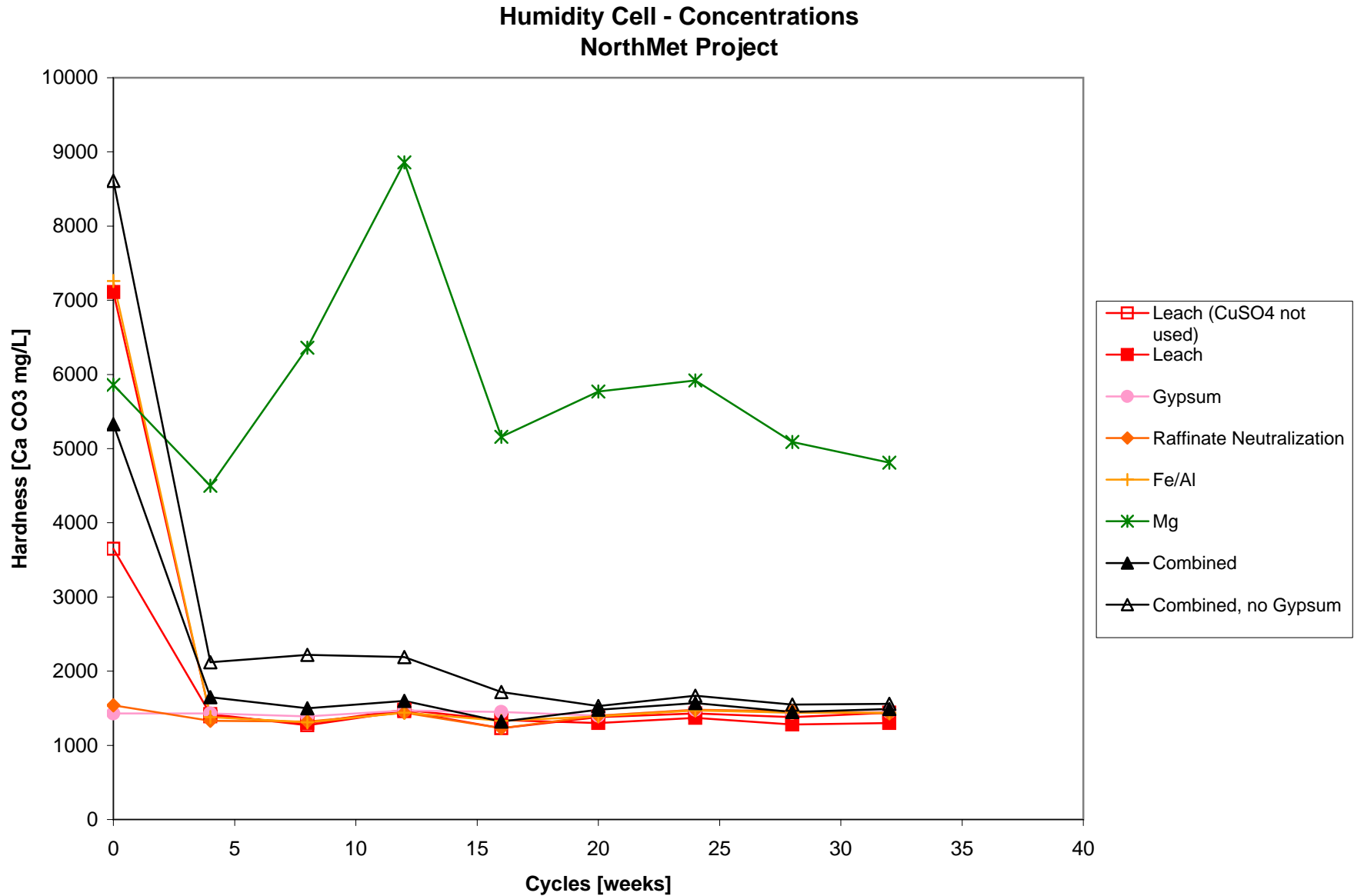
Humidity Cell - Concentrations
NorthMet Project

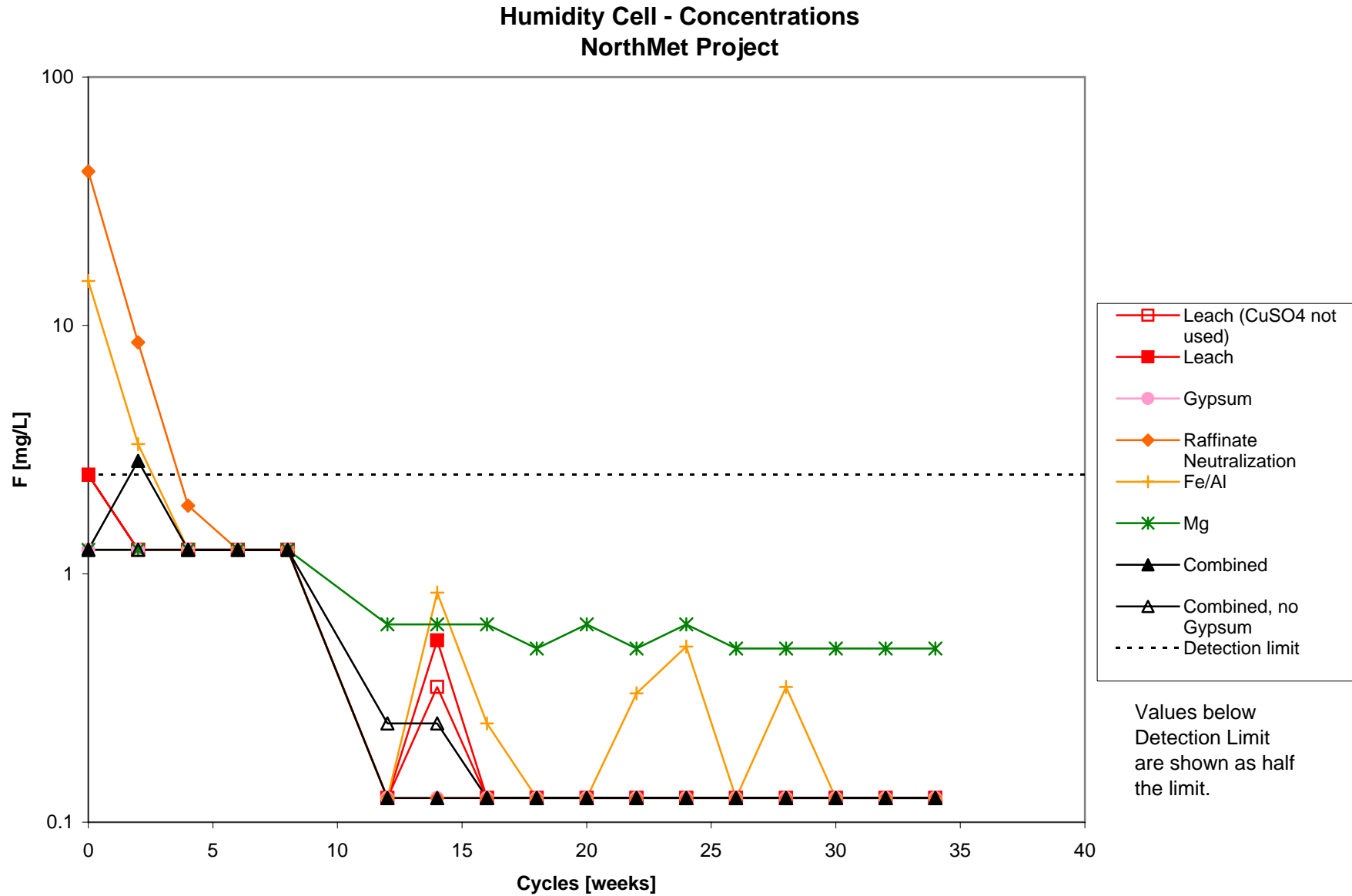


Humidity Cell - Concentrations
NorthMet Project

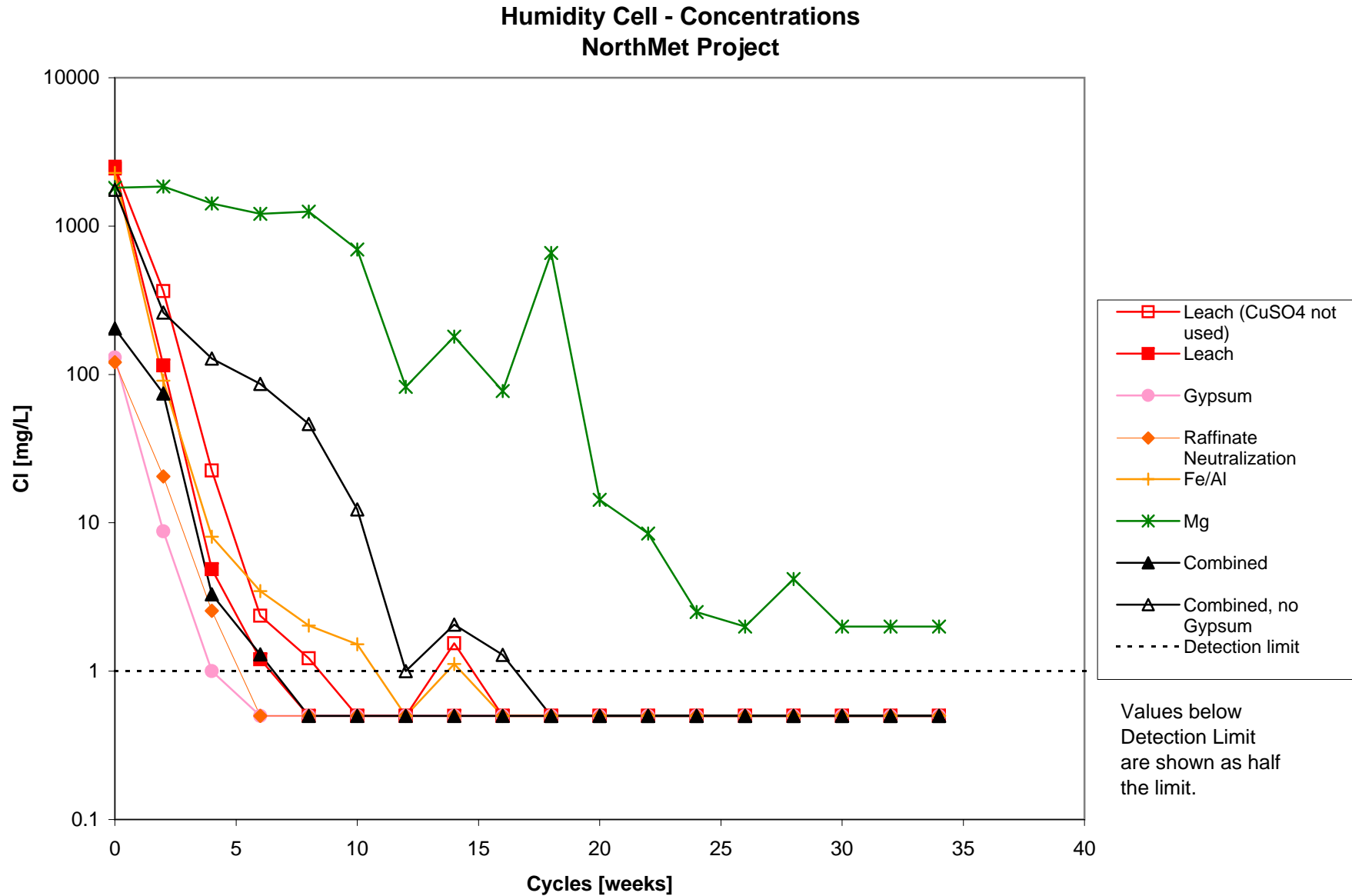




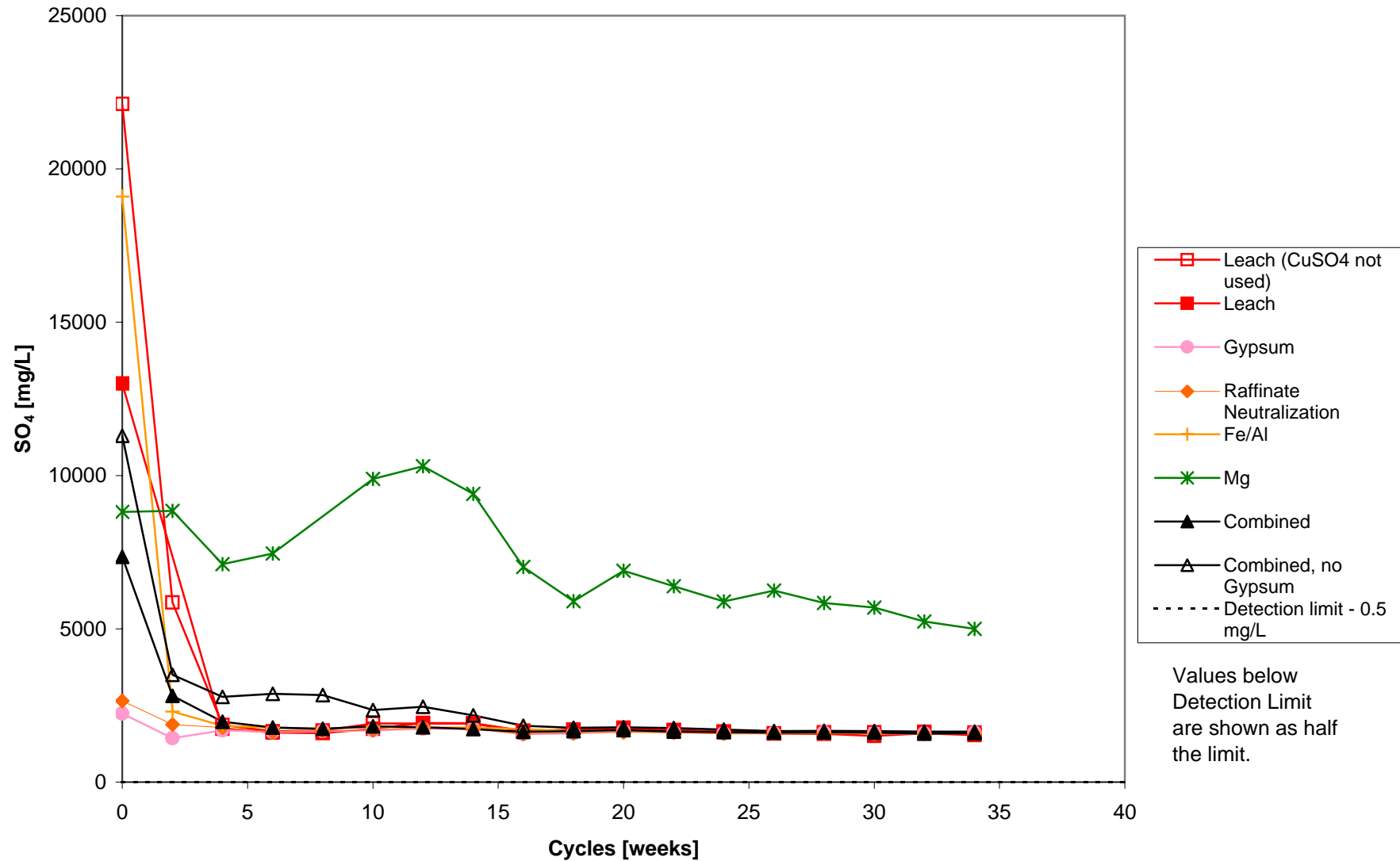




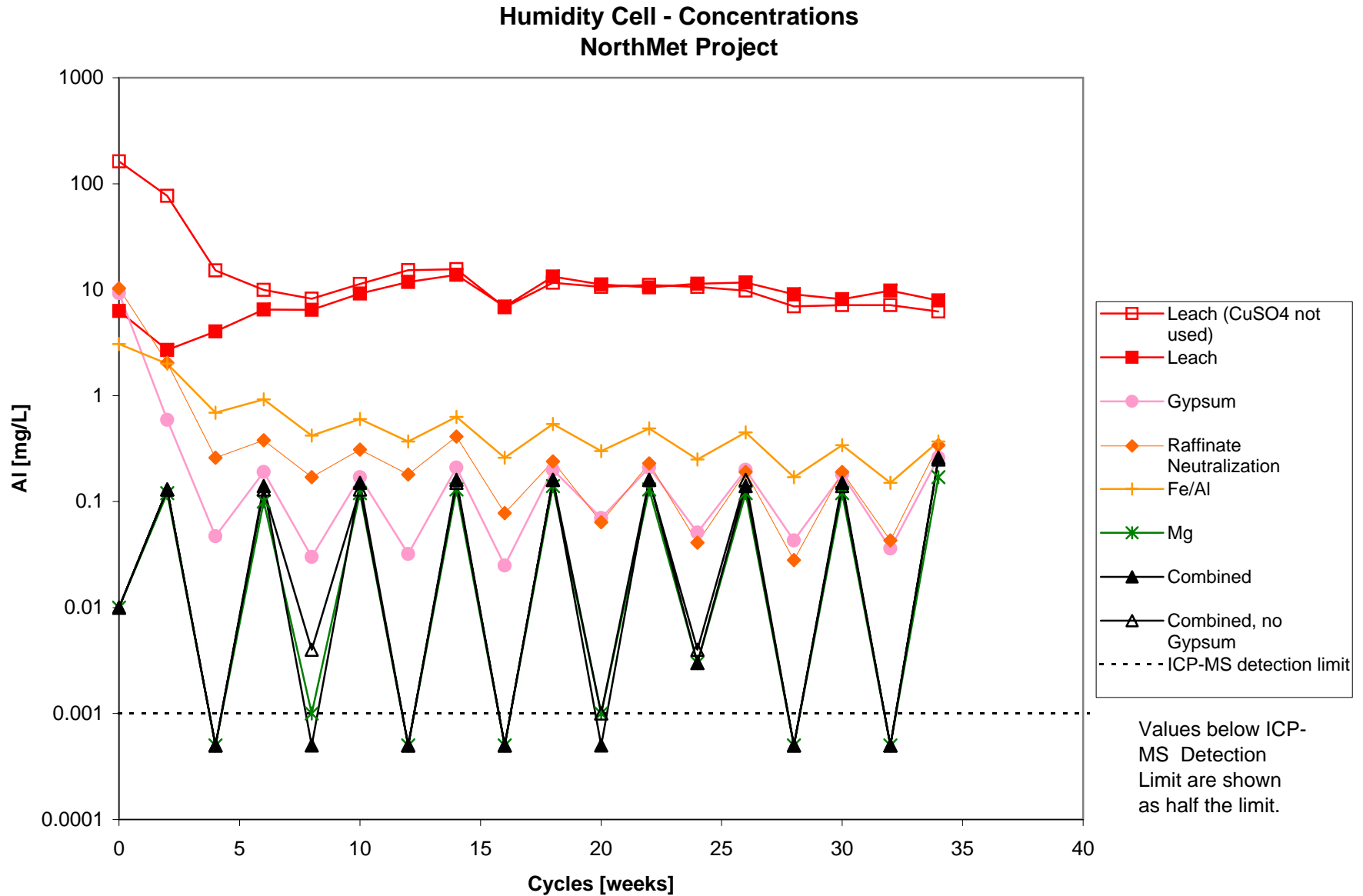
Values below
Detection Limit
are shown as half
the limit.



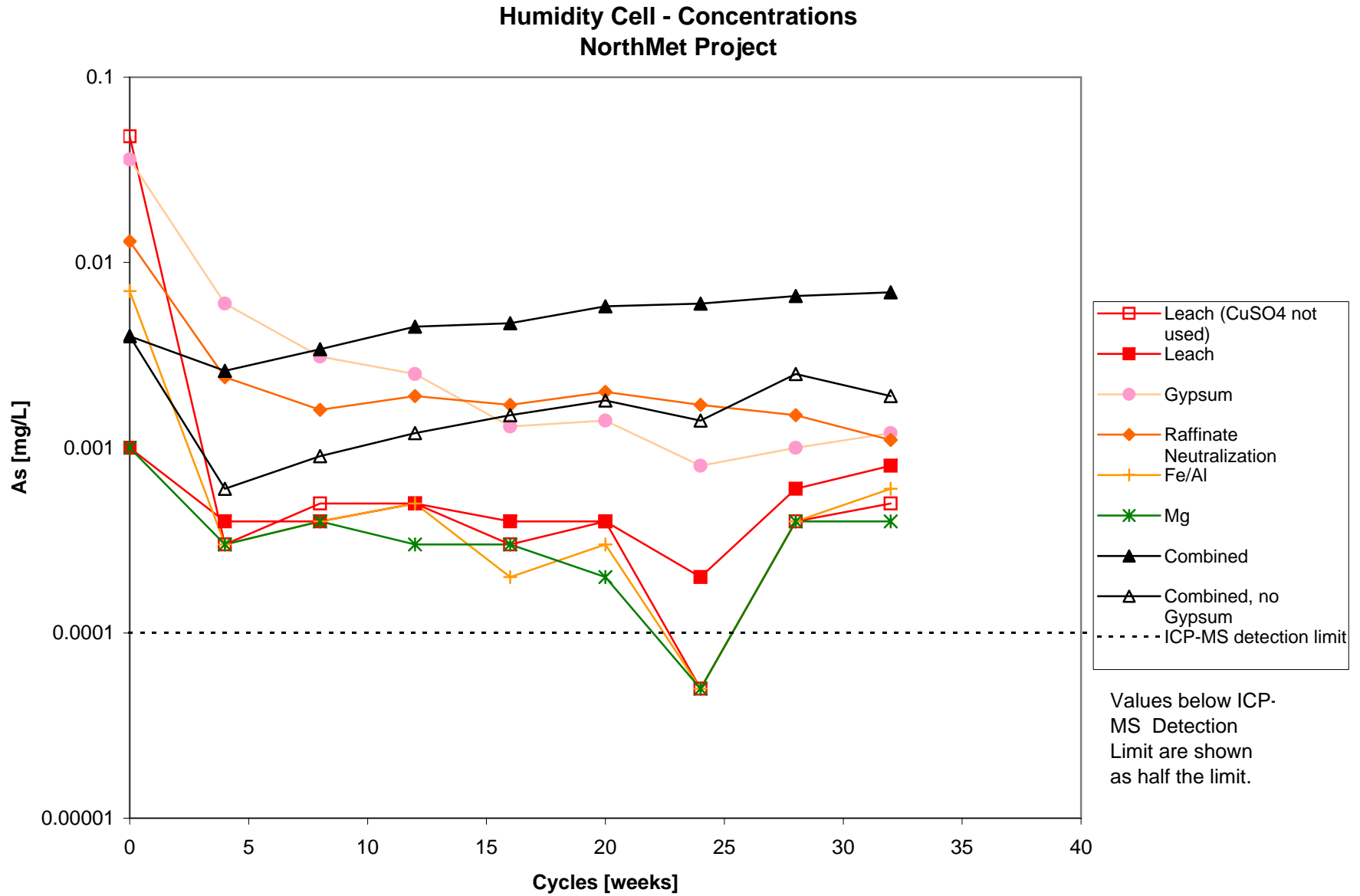
Humidity Cell - Concentrations
NorthMet Project



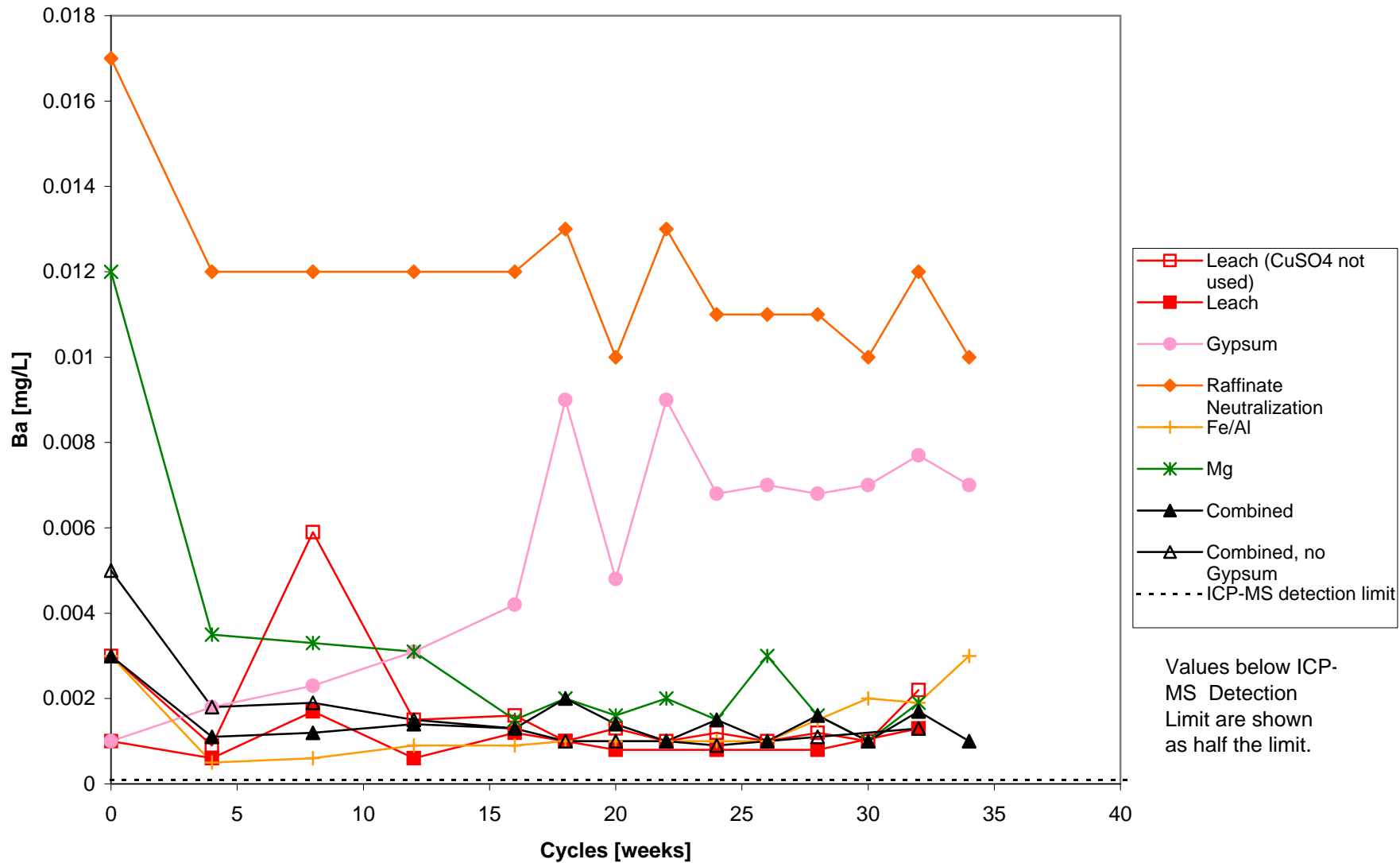
Values below
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are shown as half
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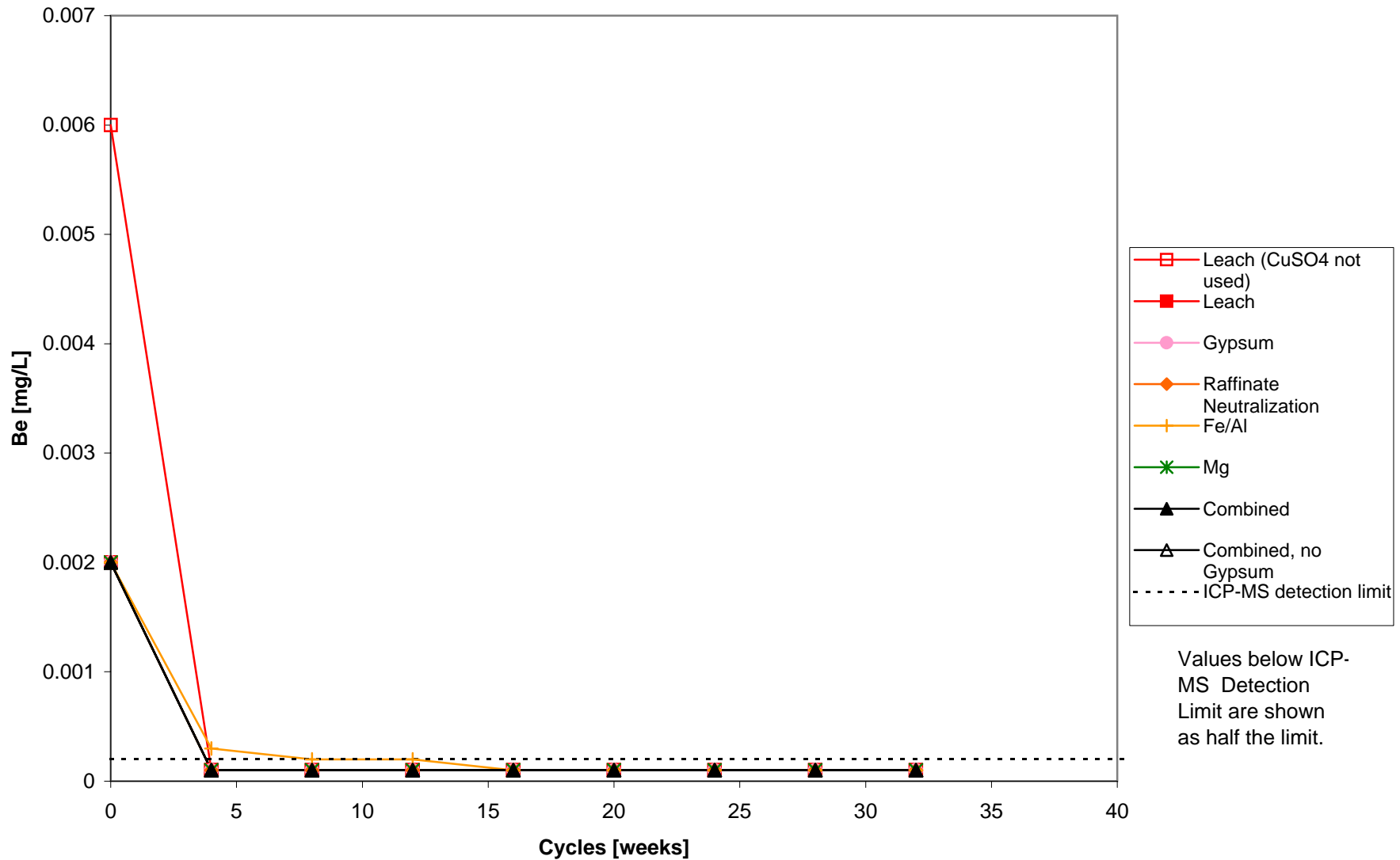
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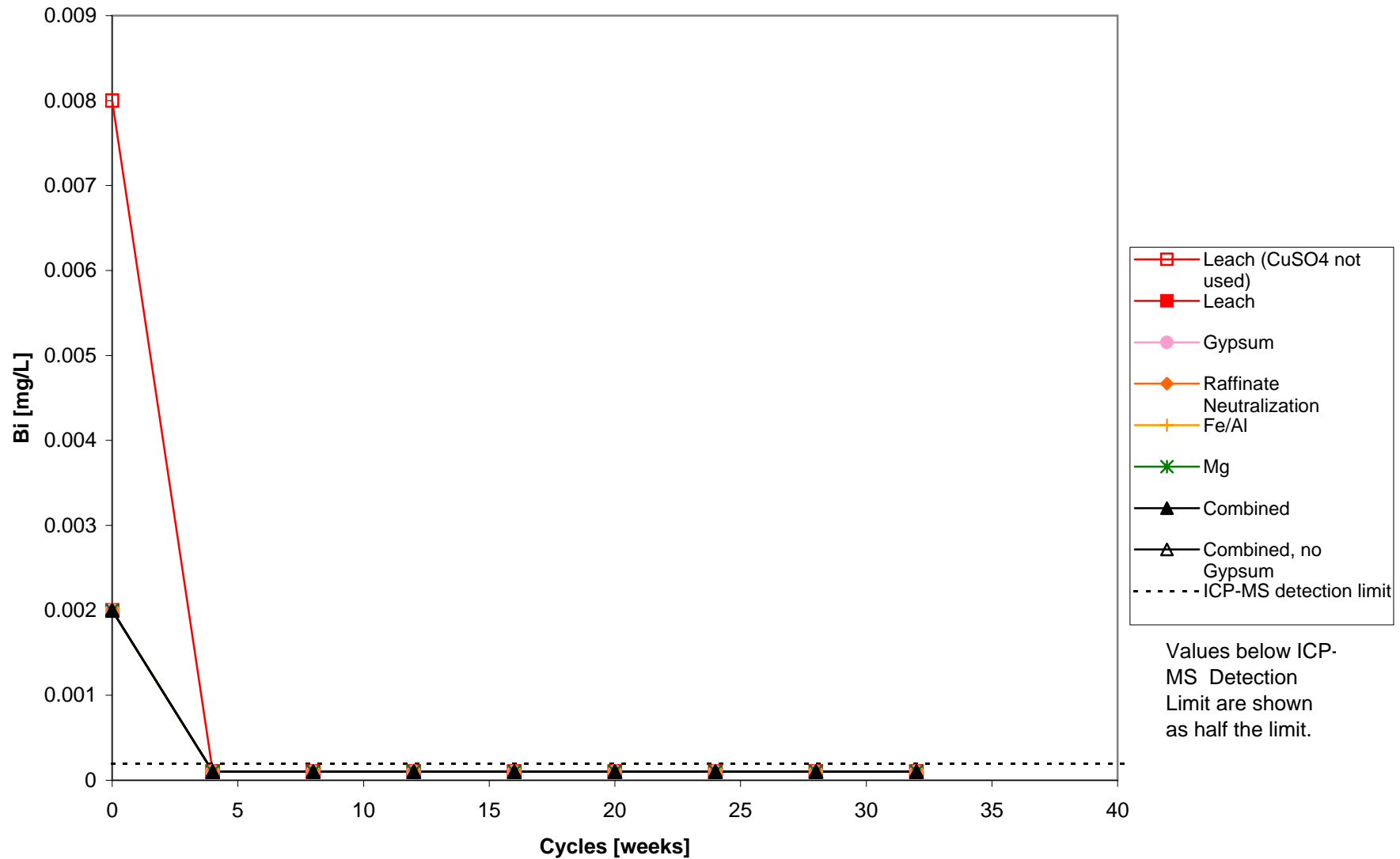
Humidity Cell - Concentrations
NorthMet Project



Humidity Cell - Concentrations
NorthMet Project

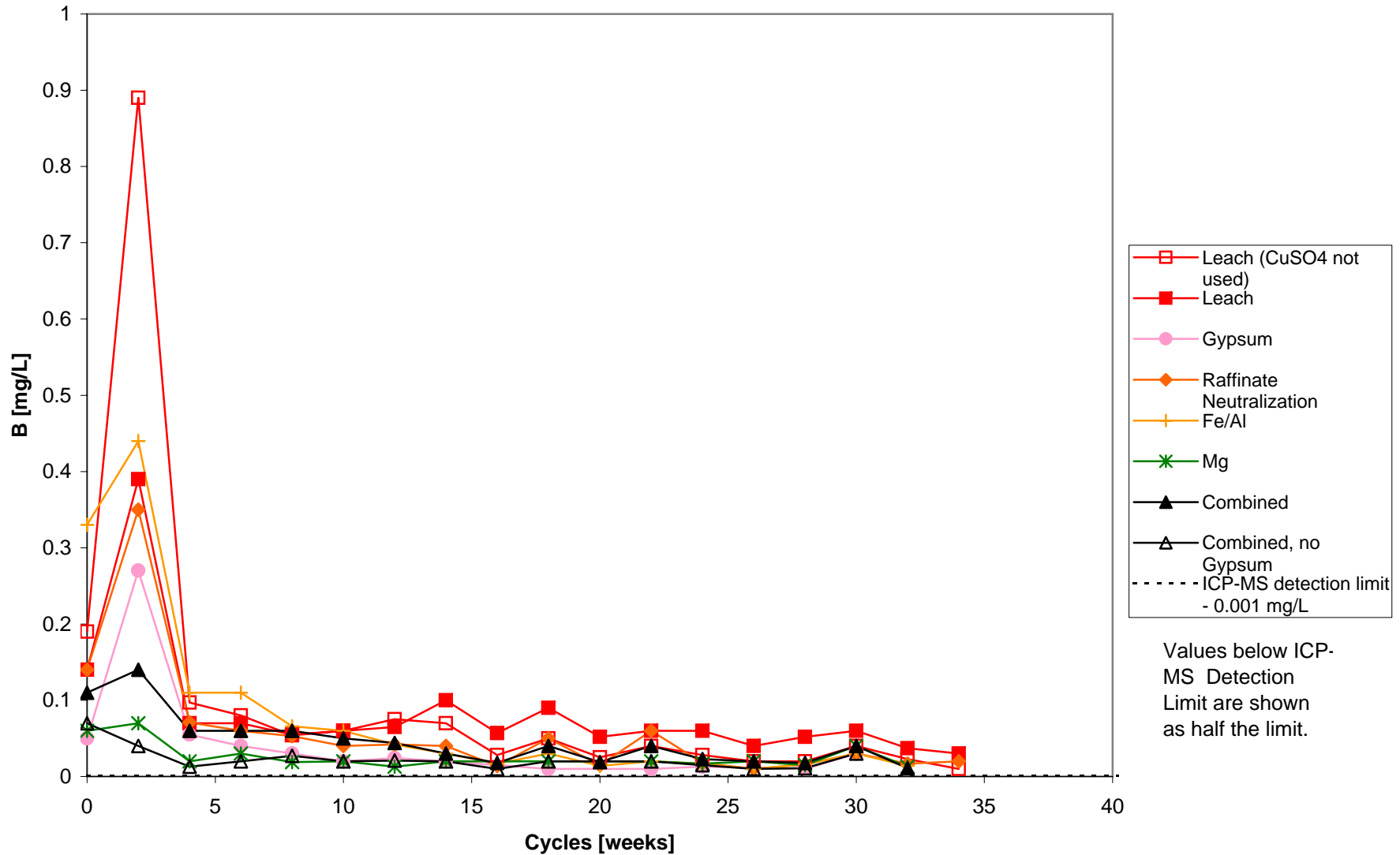


Humidity Cell - Concentrations
NorthMet Project

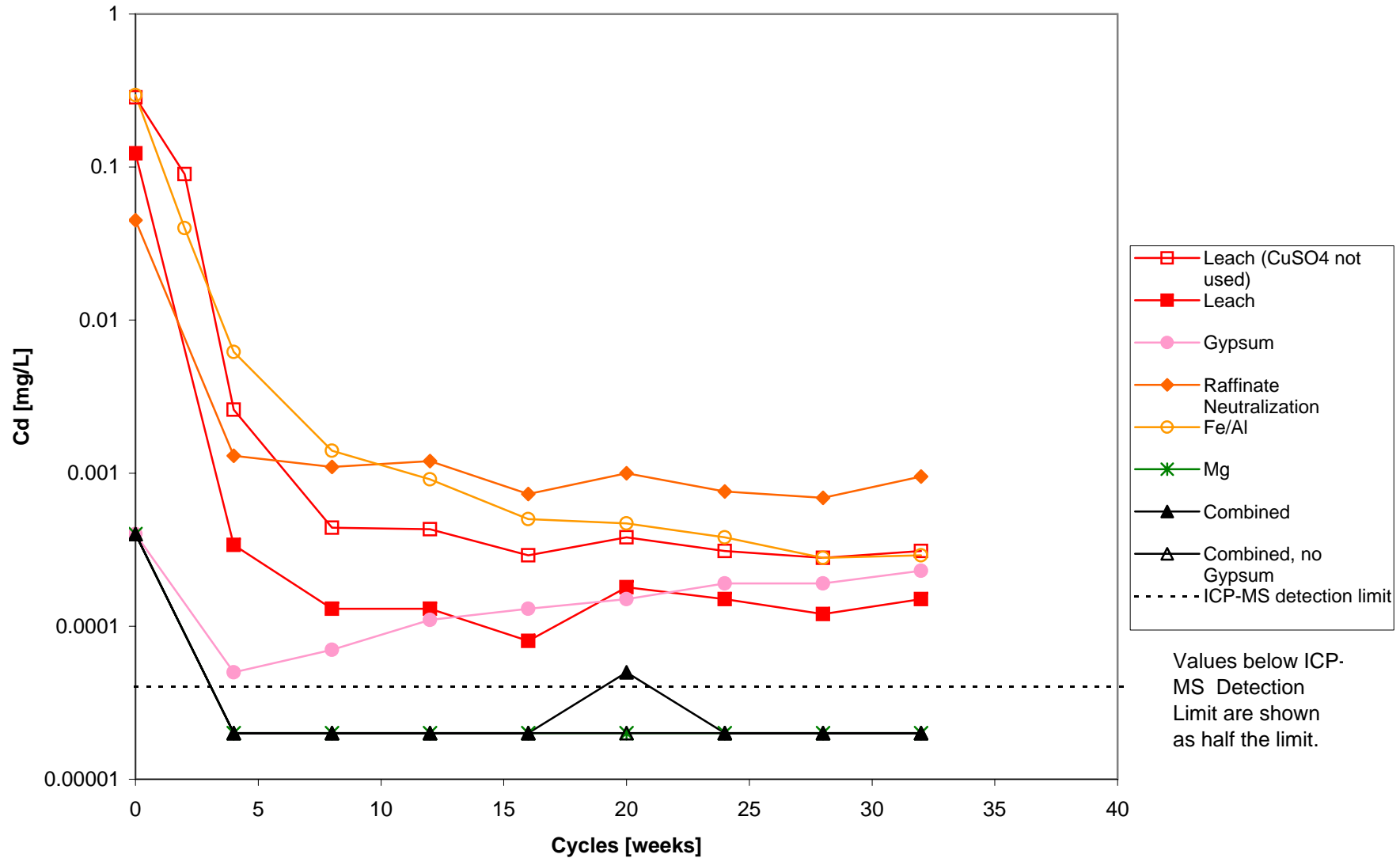


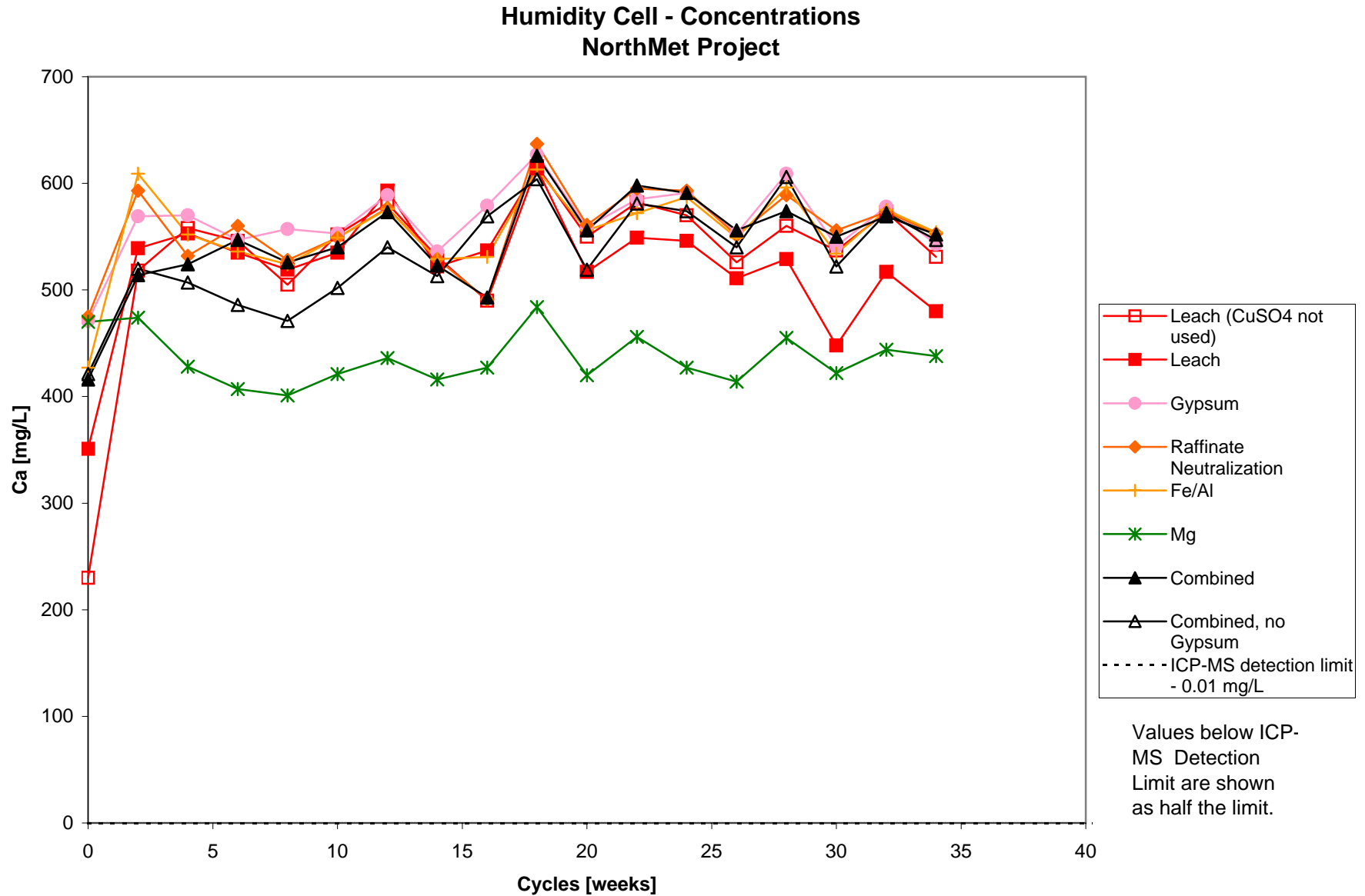
Values below ICP-MS Detection Limit are shown as half the limit.

Humidity Cell - Concentrations
NorthMet Project

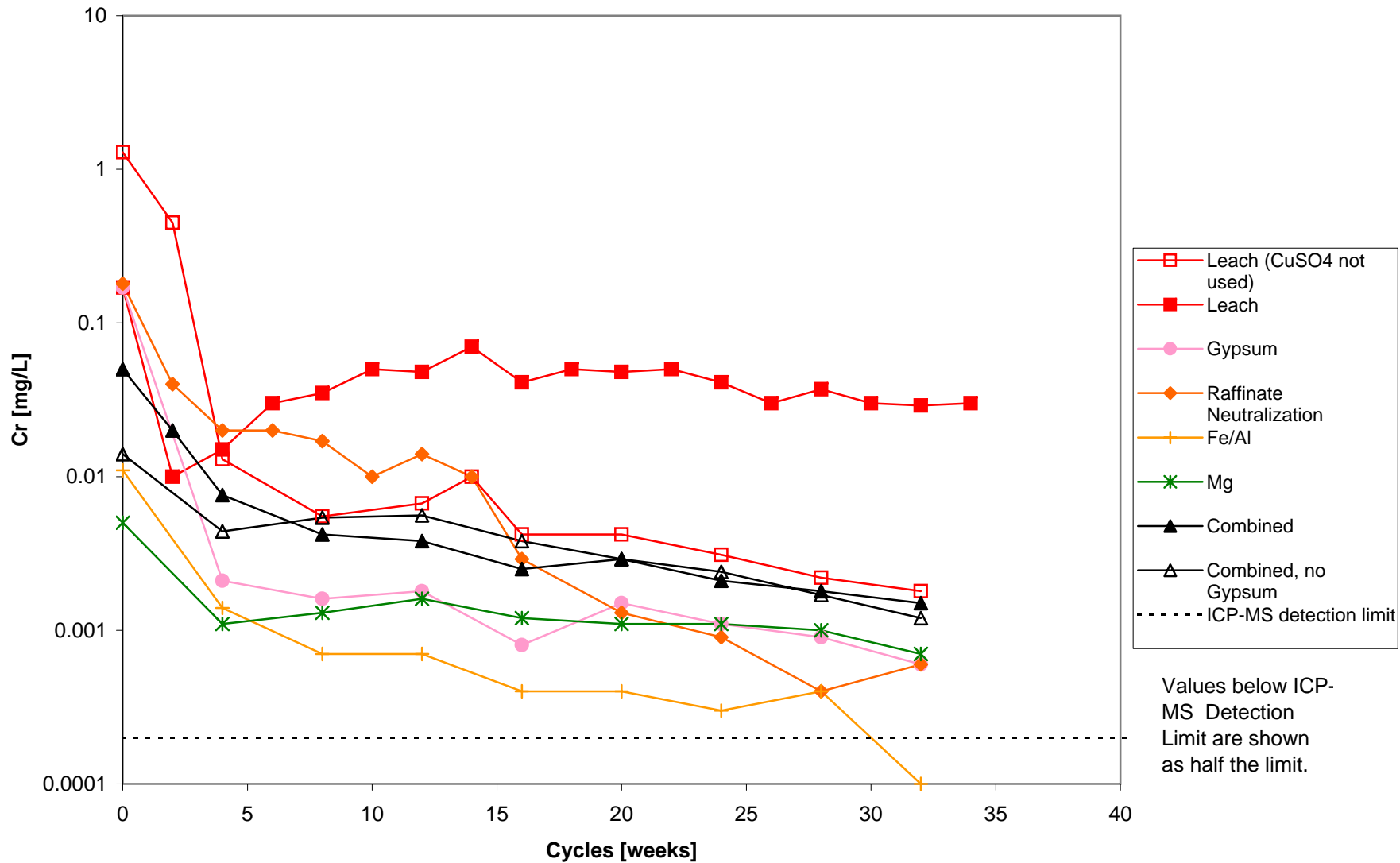


Humidity Cell - Concentrations
NorthMet Project

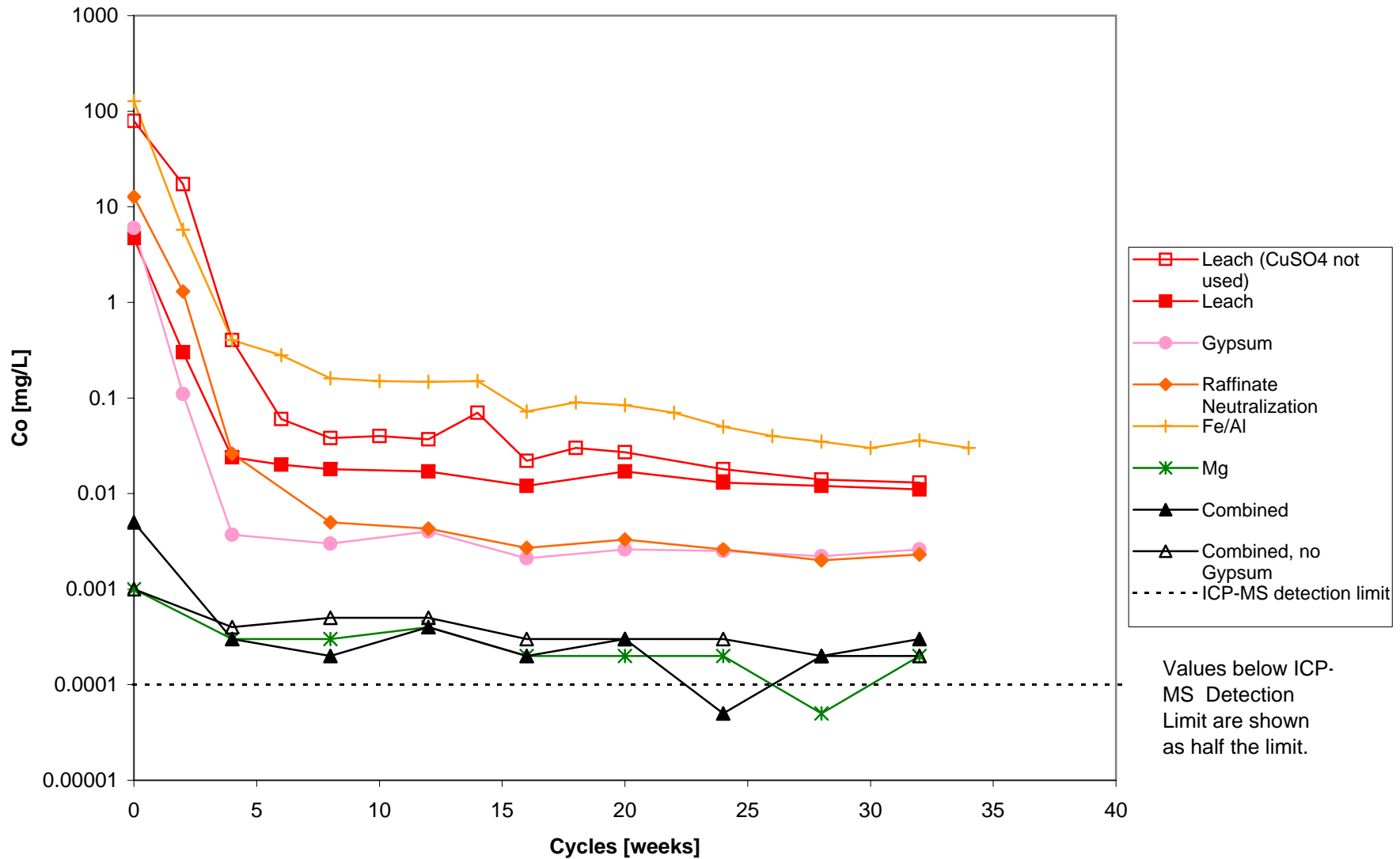




Humidity Cell - Concentrations
NorthMet Project

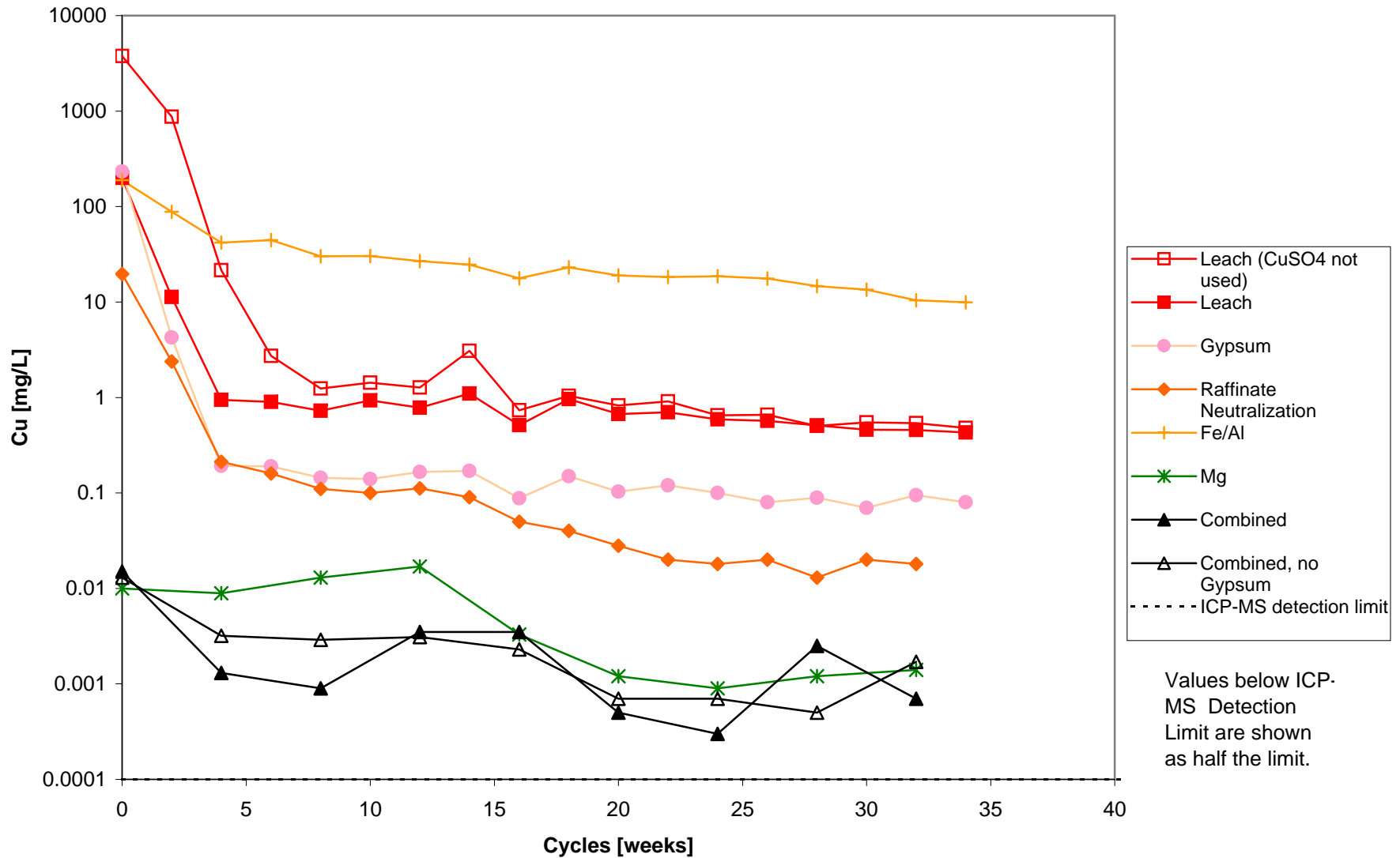


Humidity Cell - Concentrations
NorthMet Project



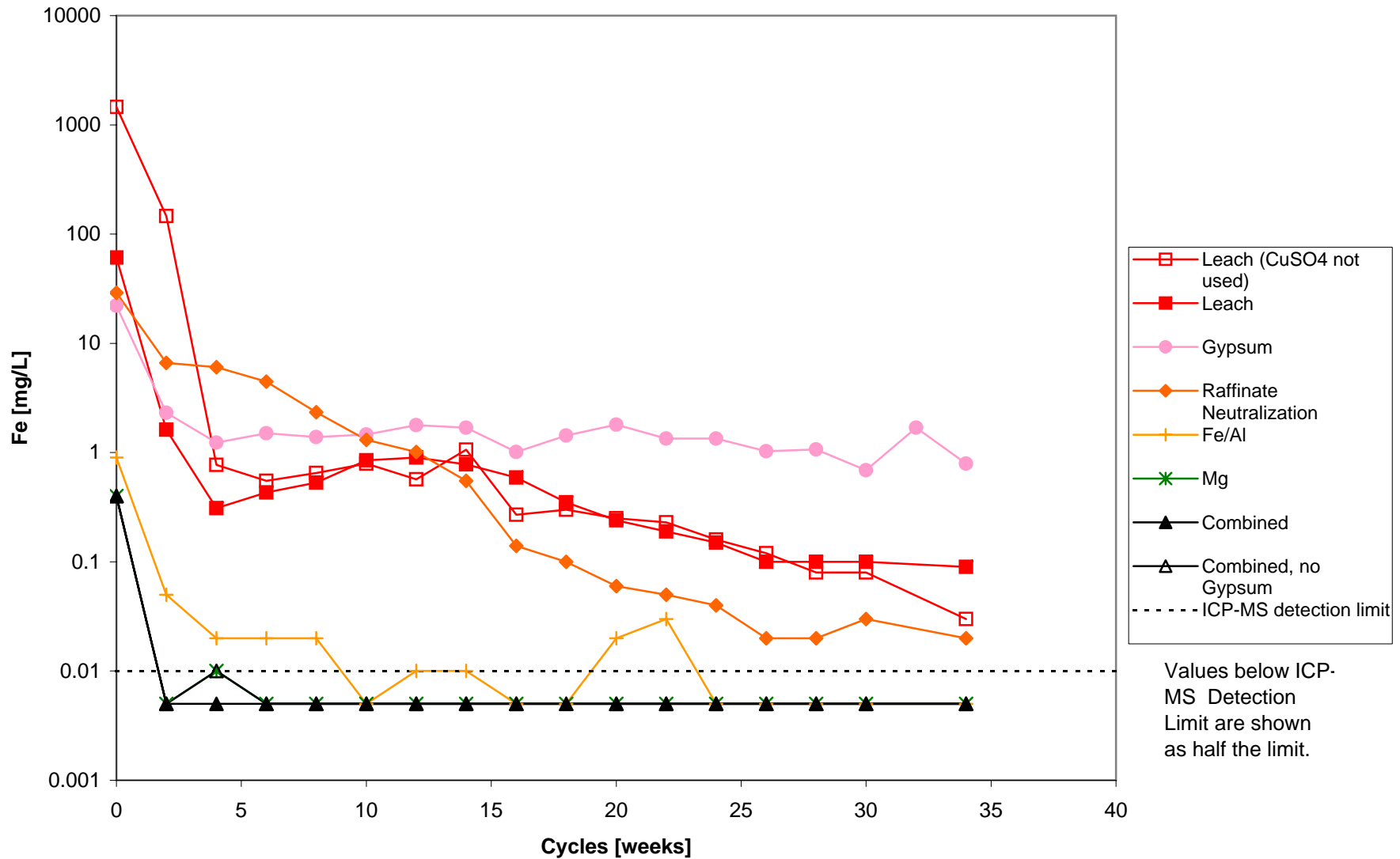
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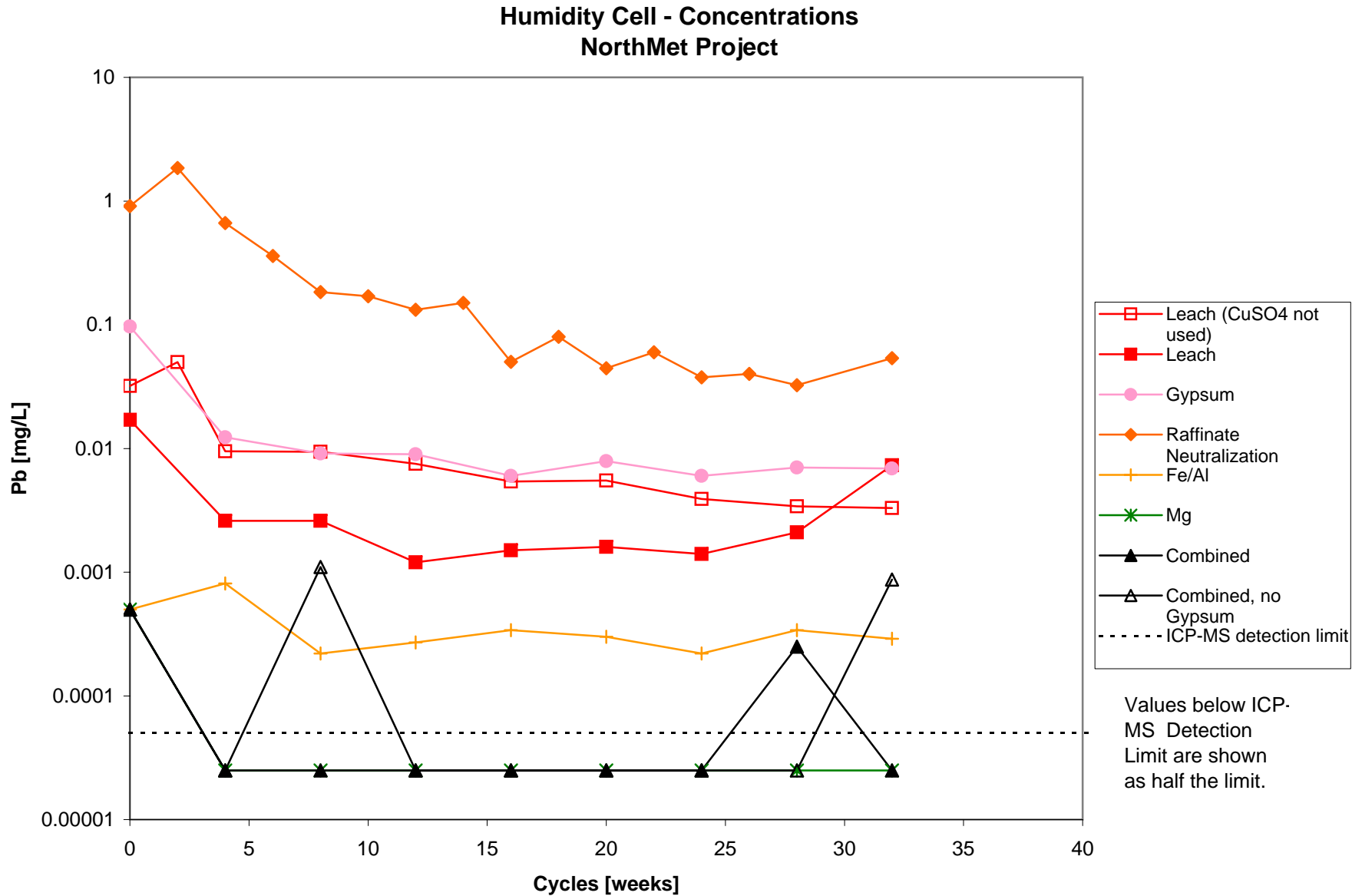
Humidity Cell - Concentrations
NorthMet Project

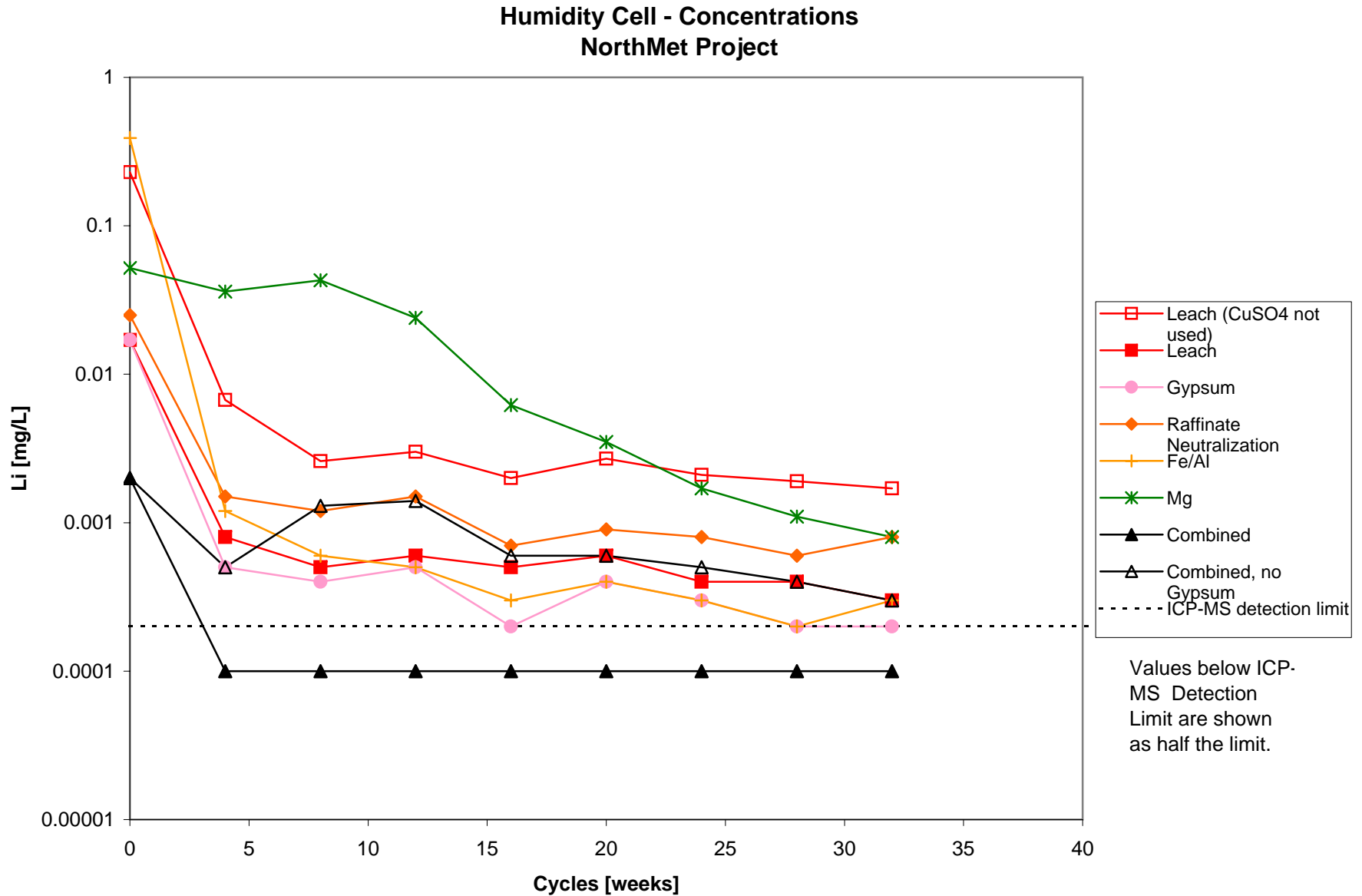


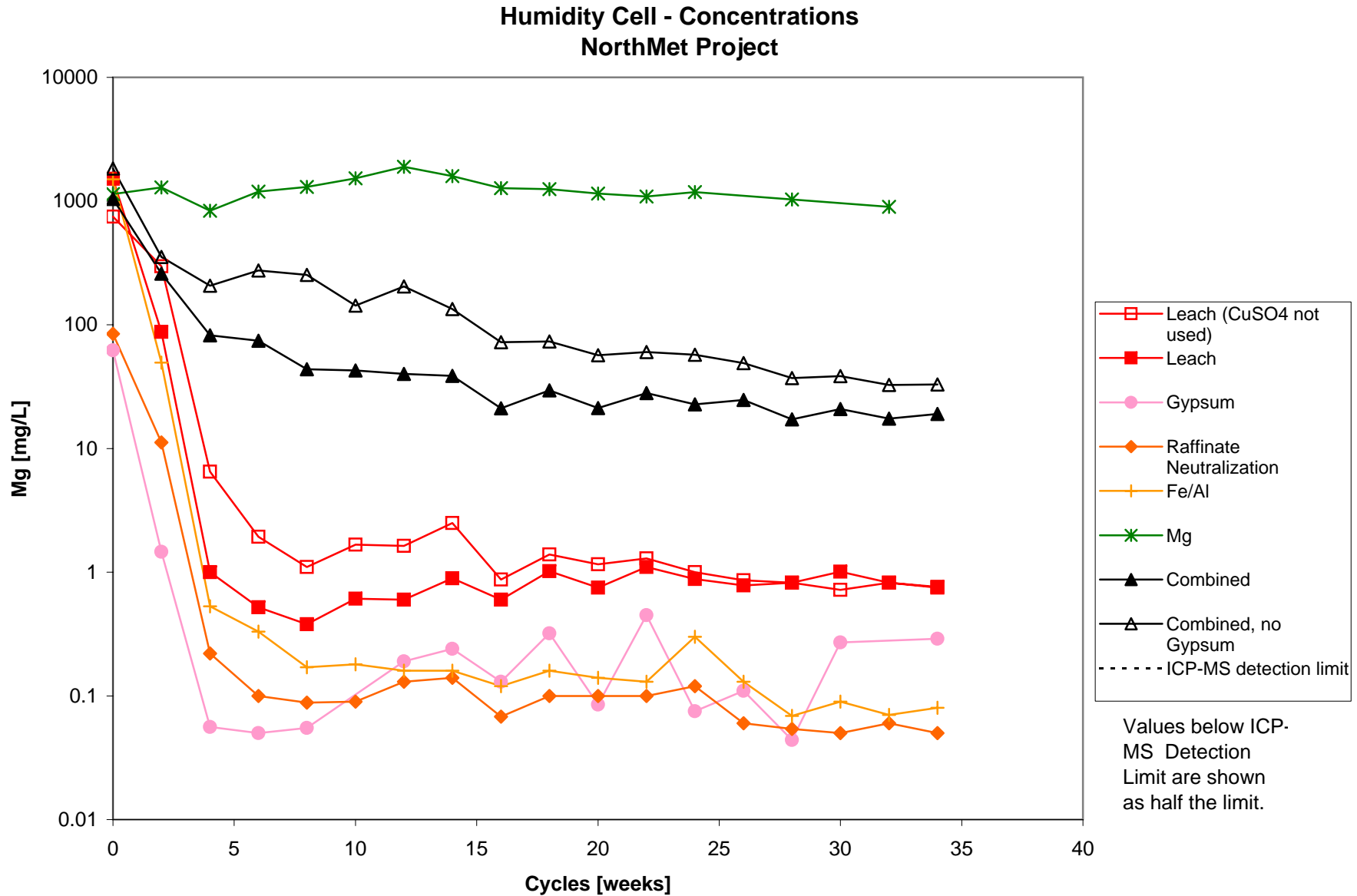
Values below ICP-MS Detection Limit are shown as half the limit.

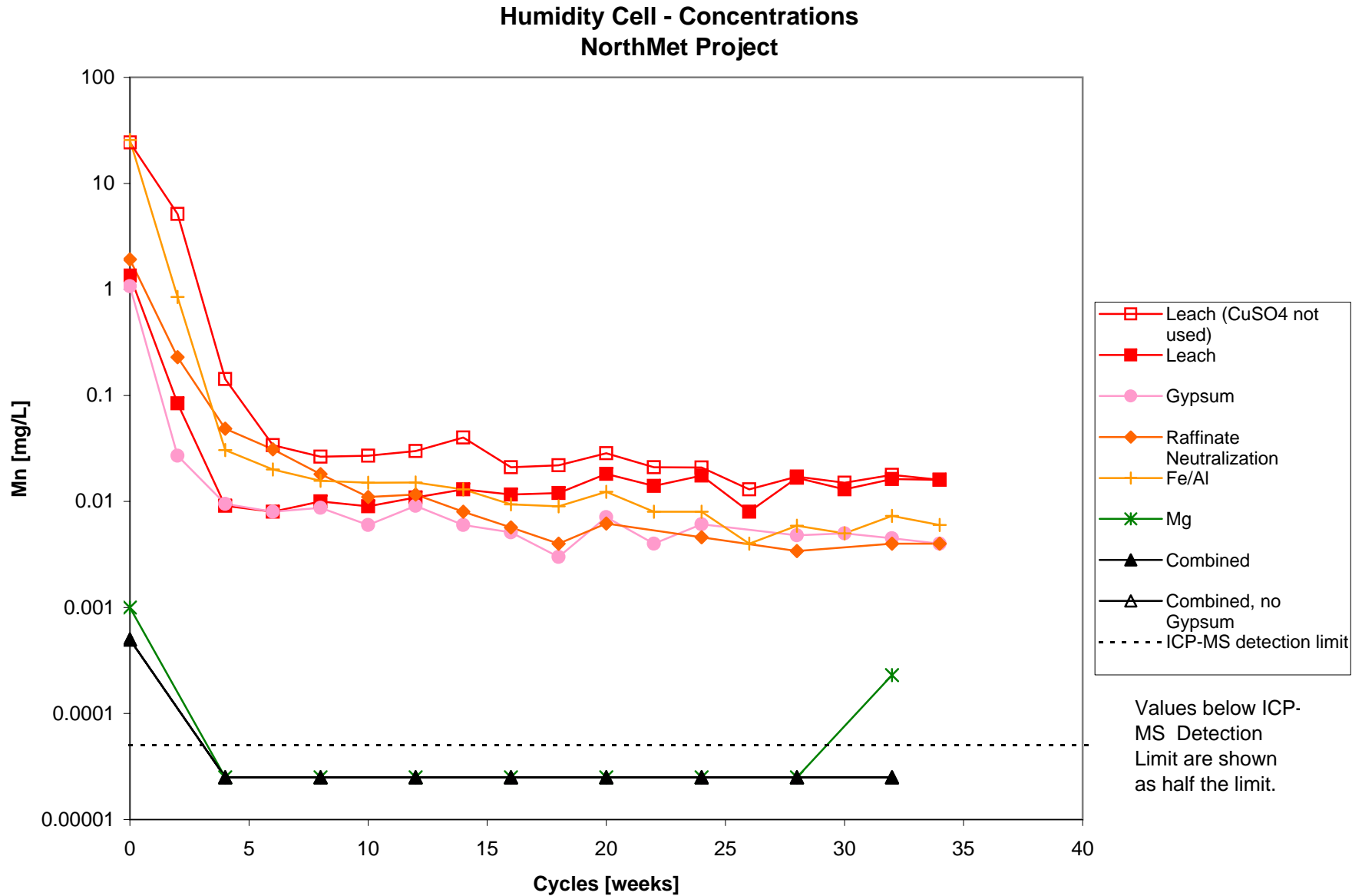
Humidity Cell - Concentrations
NorthMet Project



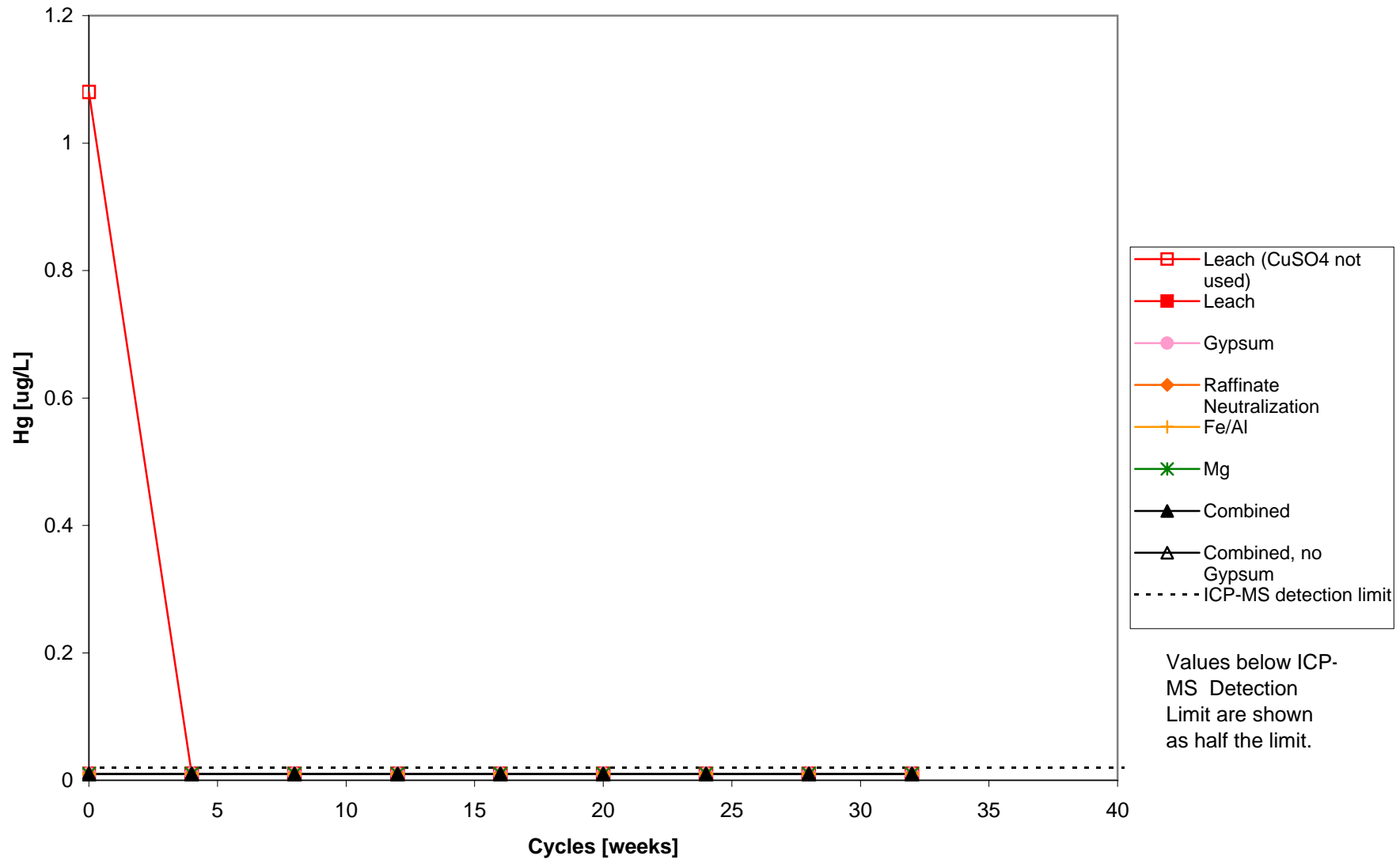


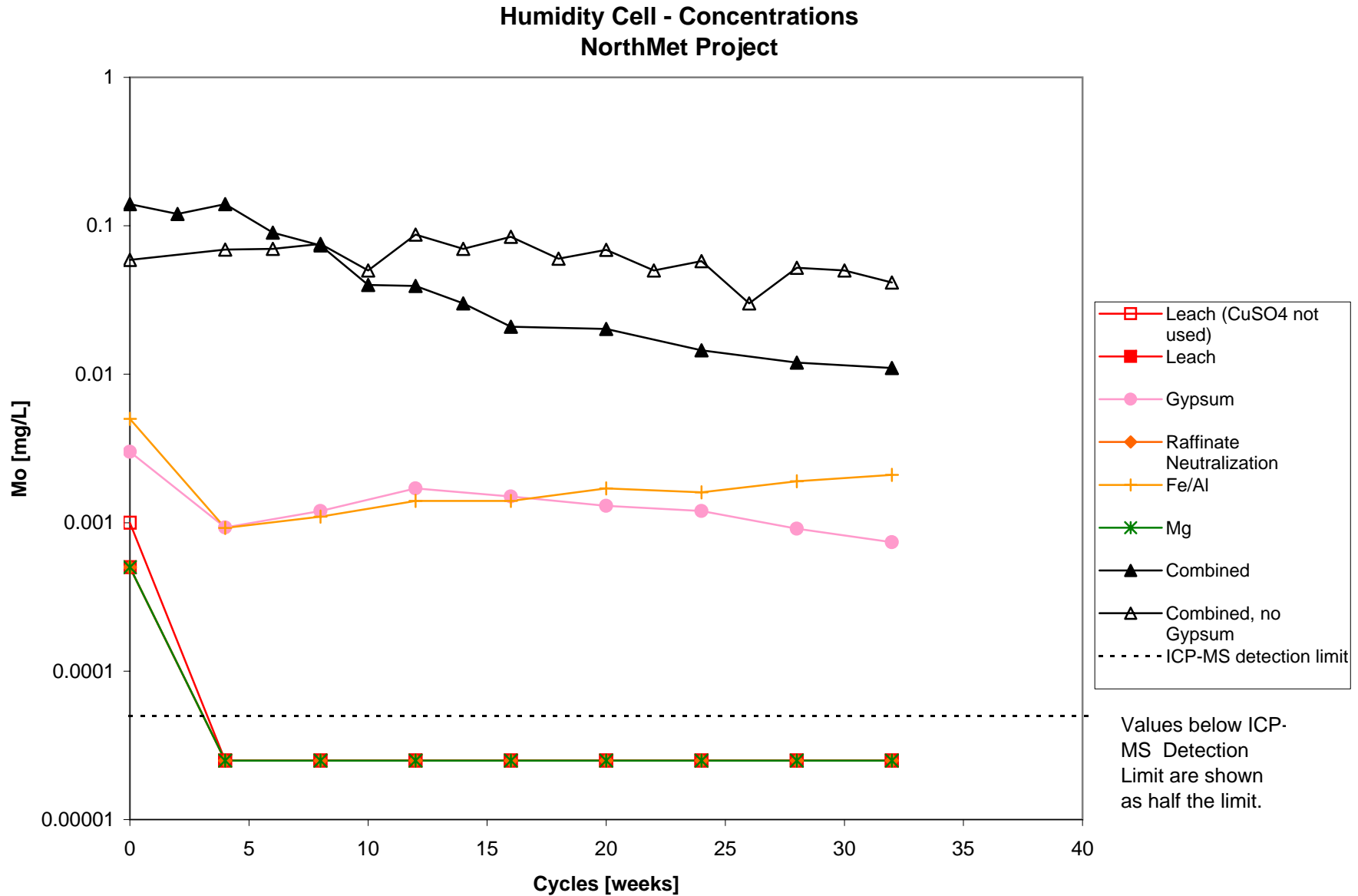




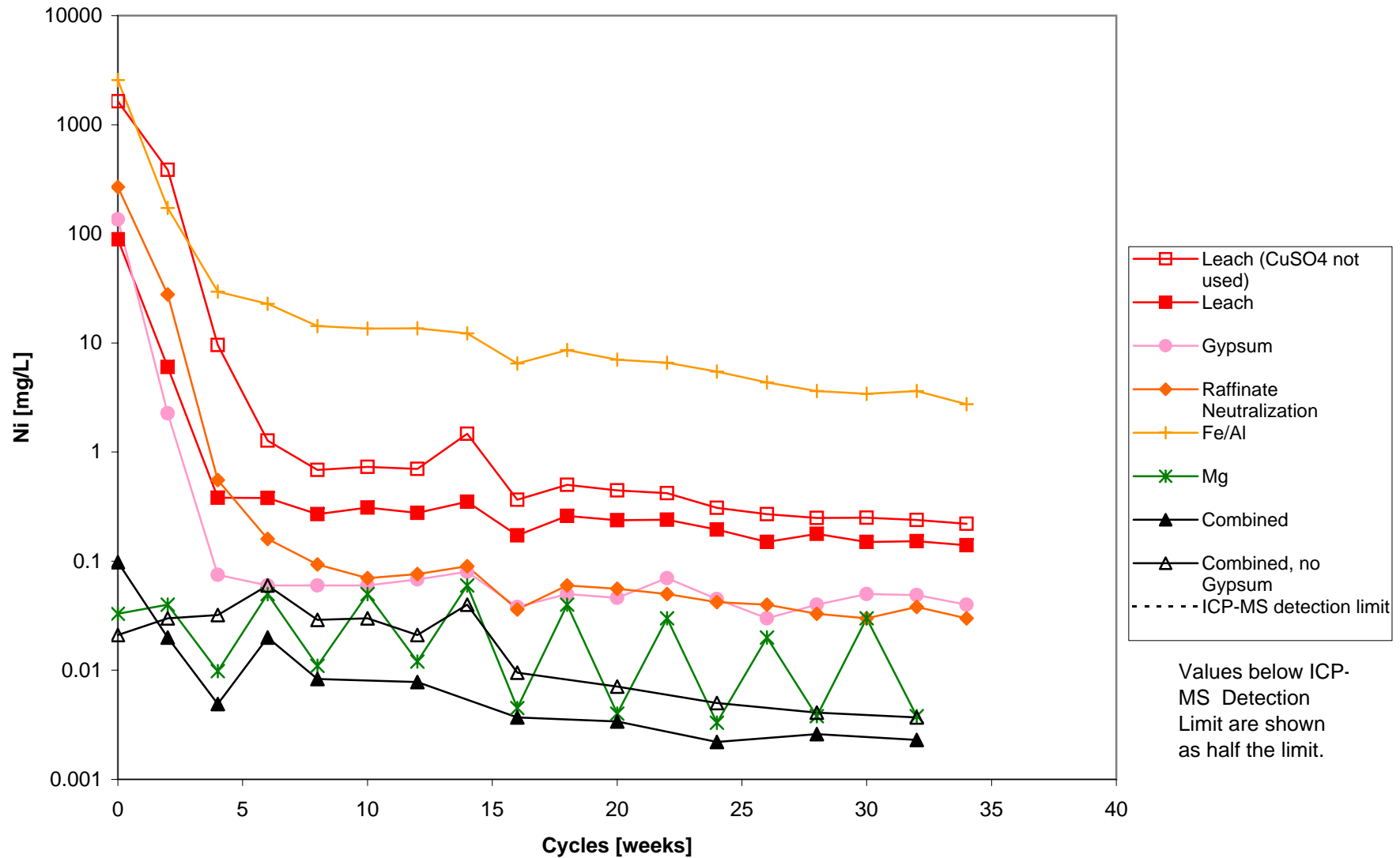


Humidity Cell - Concentrations
NorthMet Project

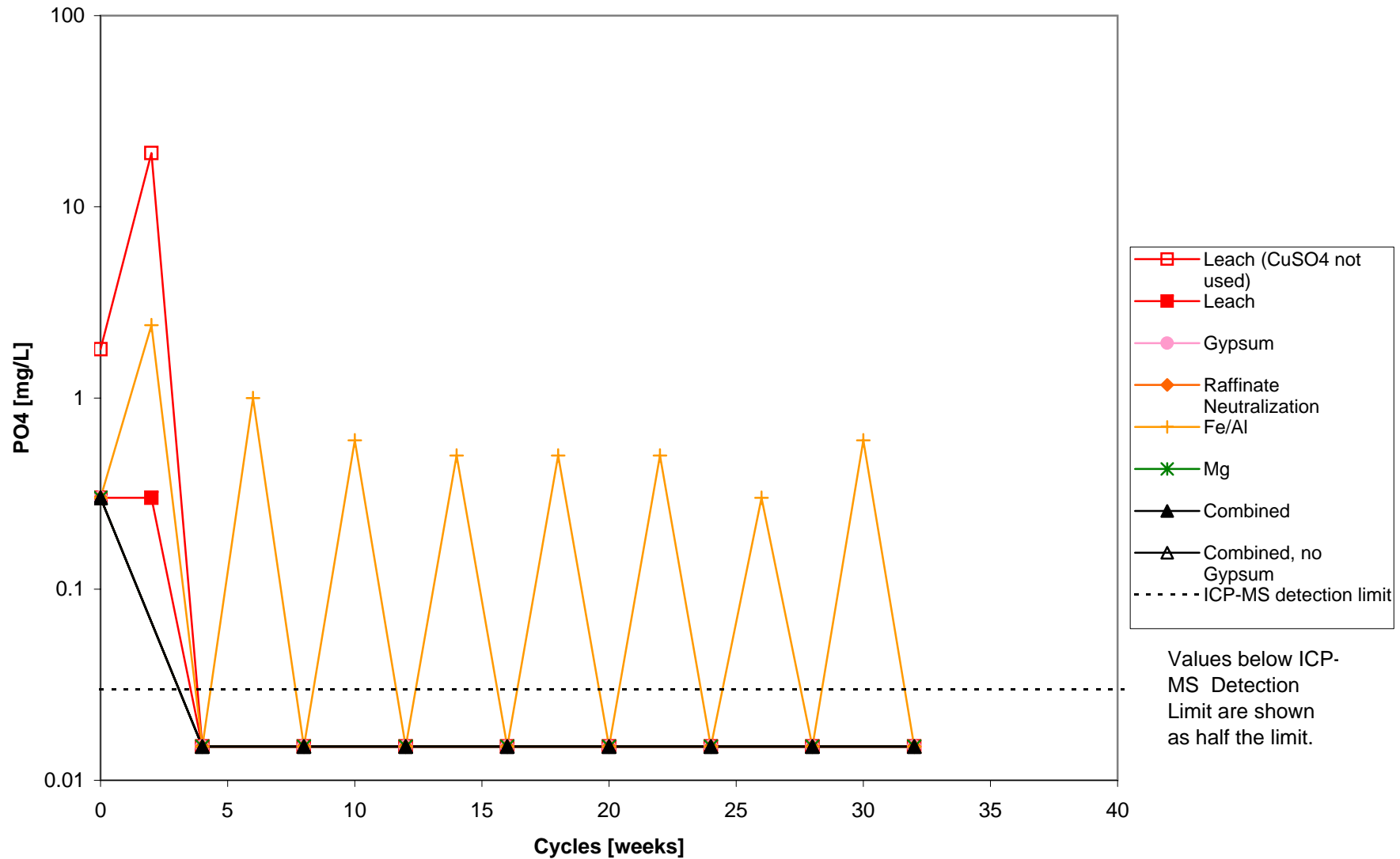




Humidity Cell - Concentrations
NorthMet Project

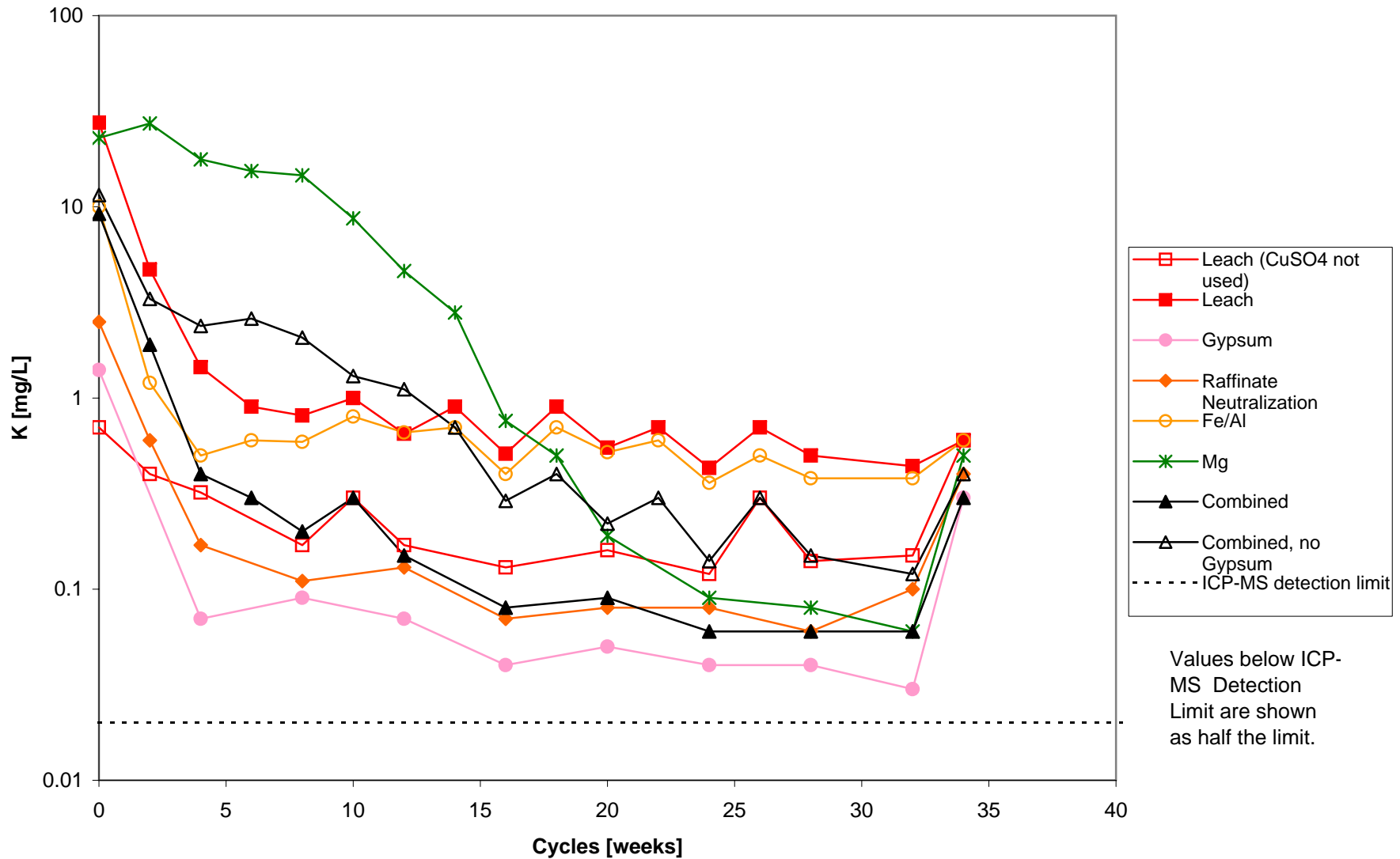


Humidity Cell - Concentrations
NorthMet Project



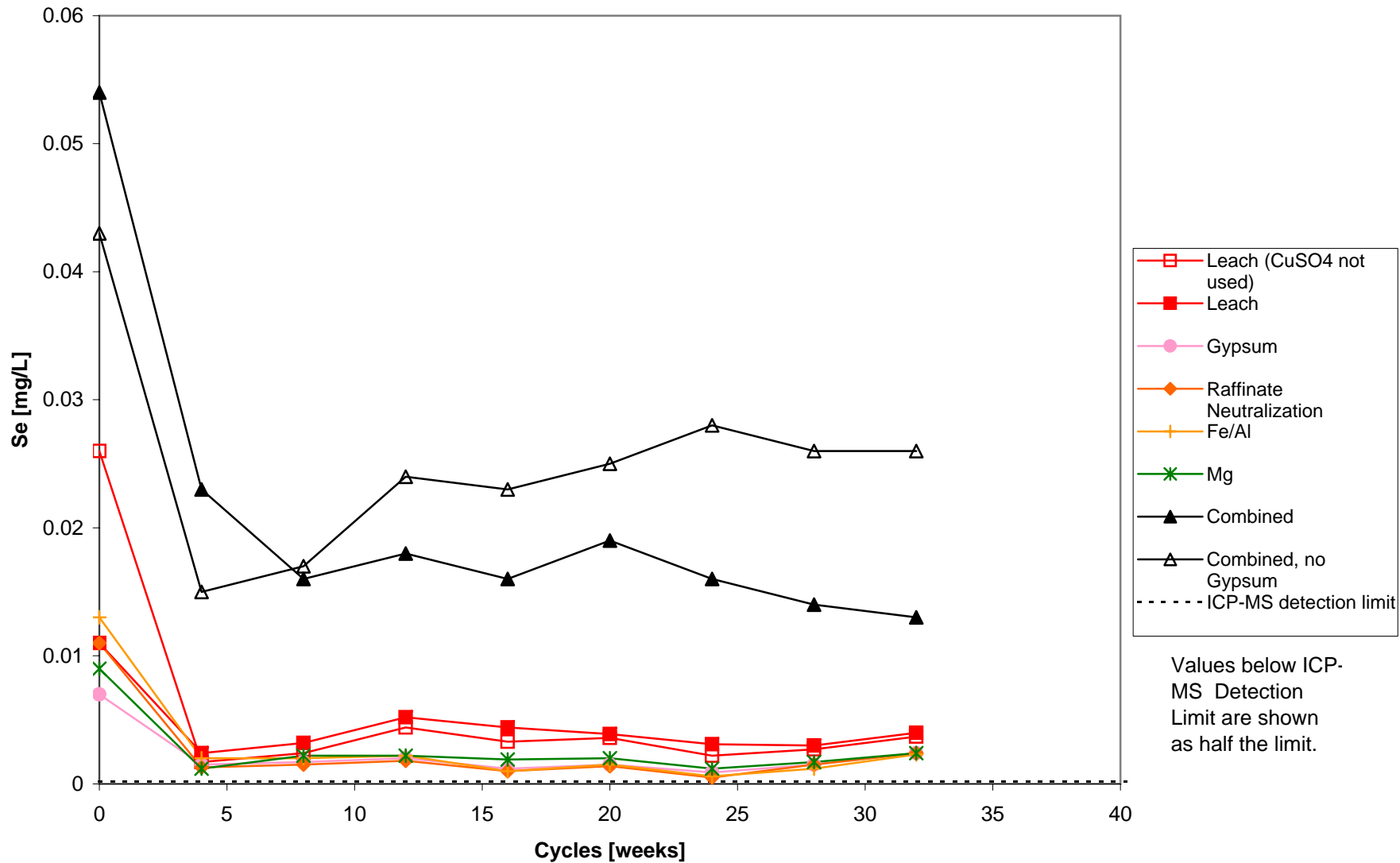
Values below ICP-MS Detection Limit are shown as half the limit.

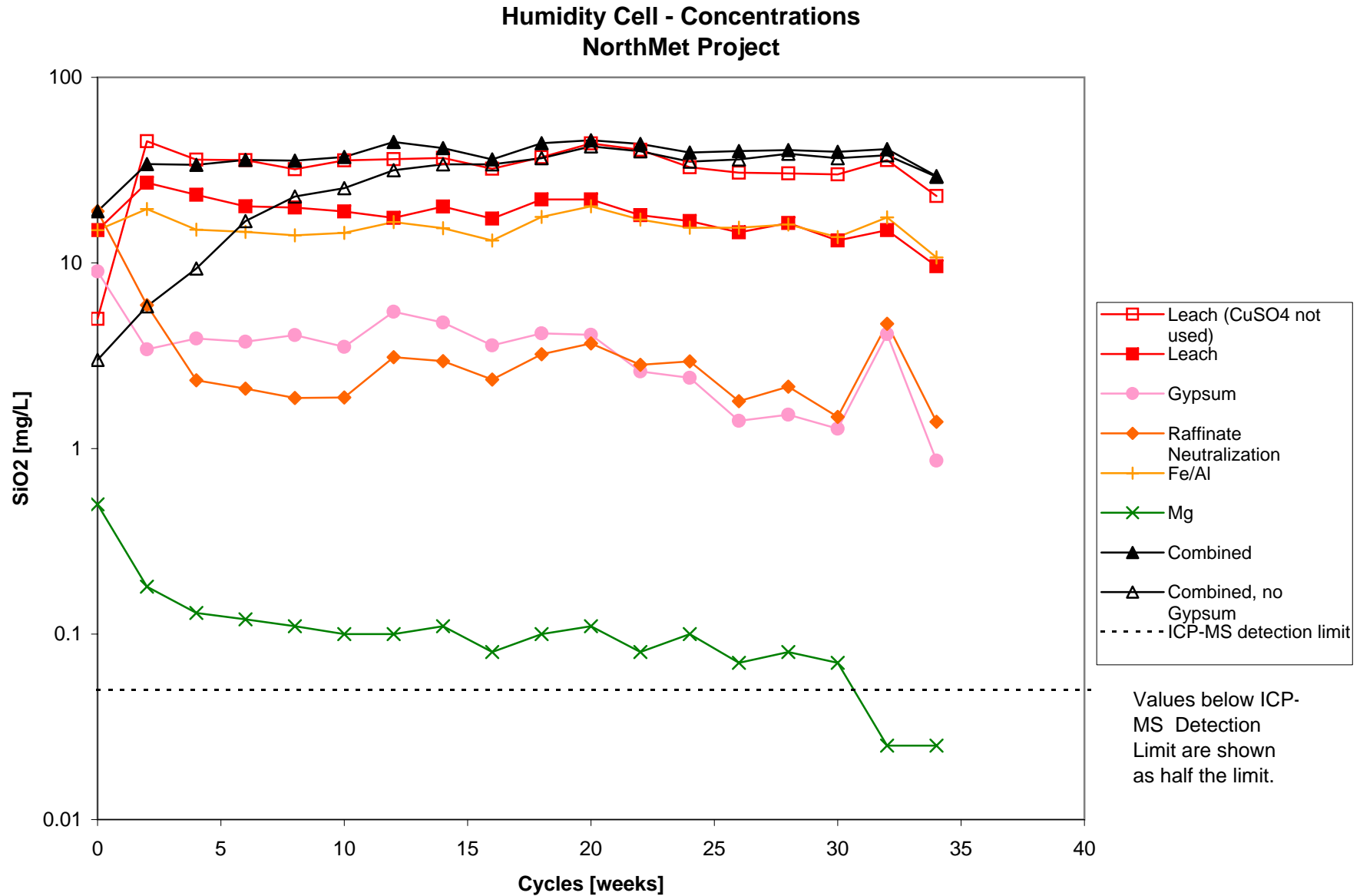
Humidity Cell - Concentrations
NorthMet Project



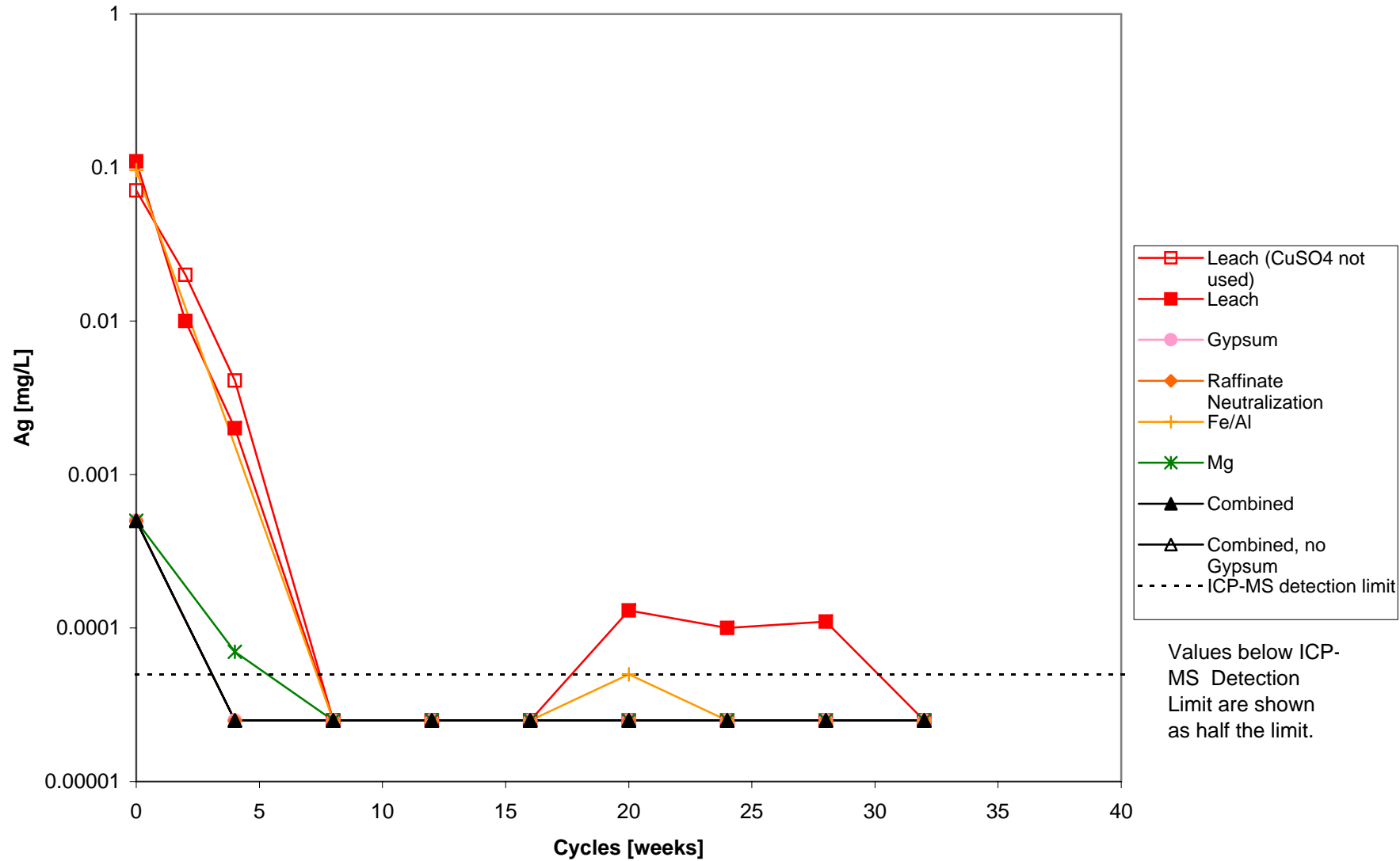
Values below ICP-MS Detection Limit are shown as half the limit.

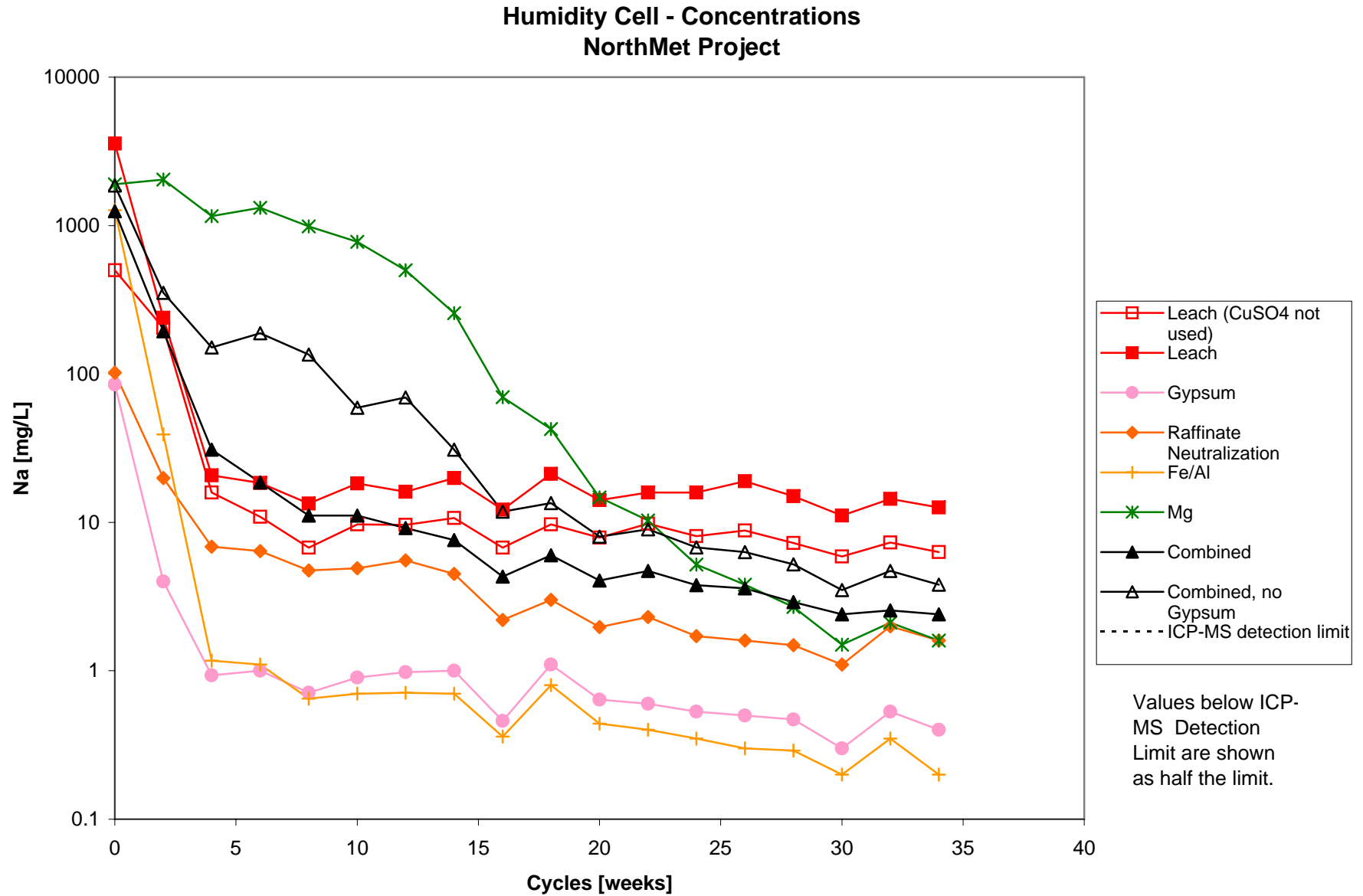
Humidity Cell - Concentrations
NorthMet Project



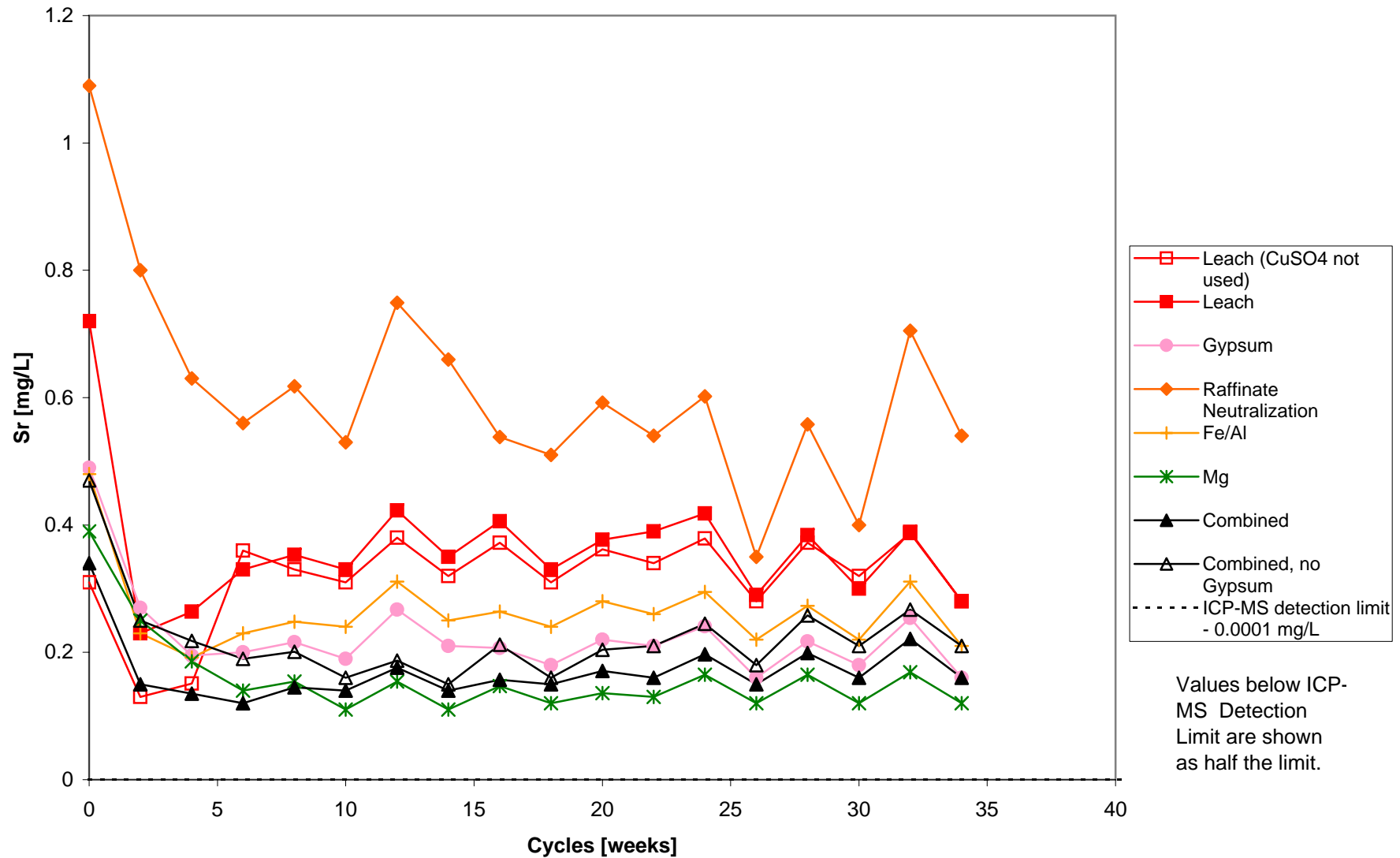


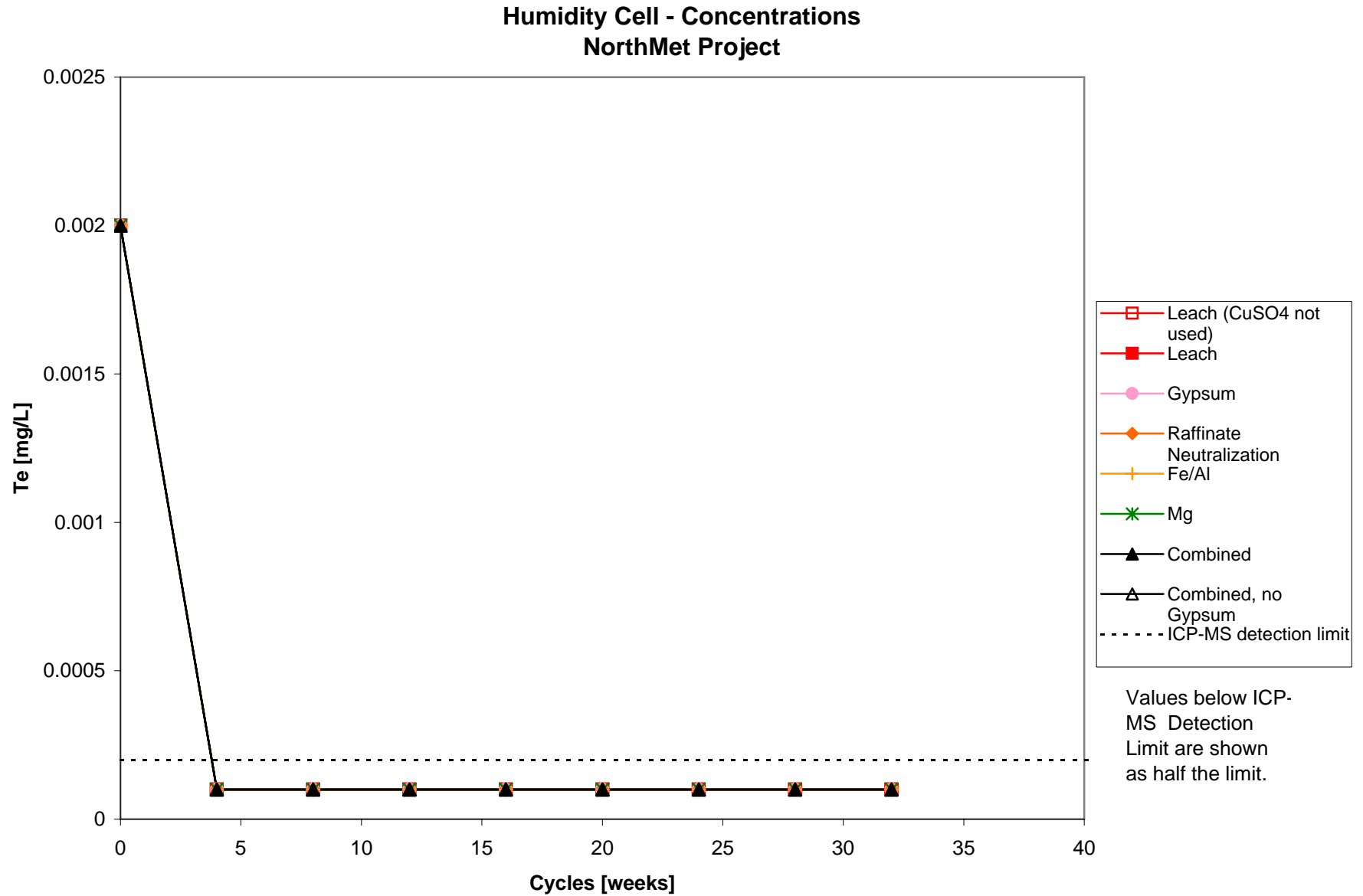
Humidity Cell - Concentrations
NorthMet Project



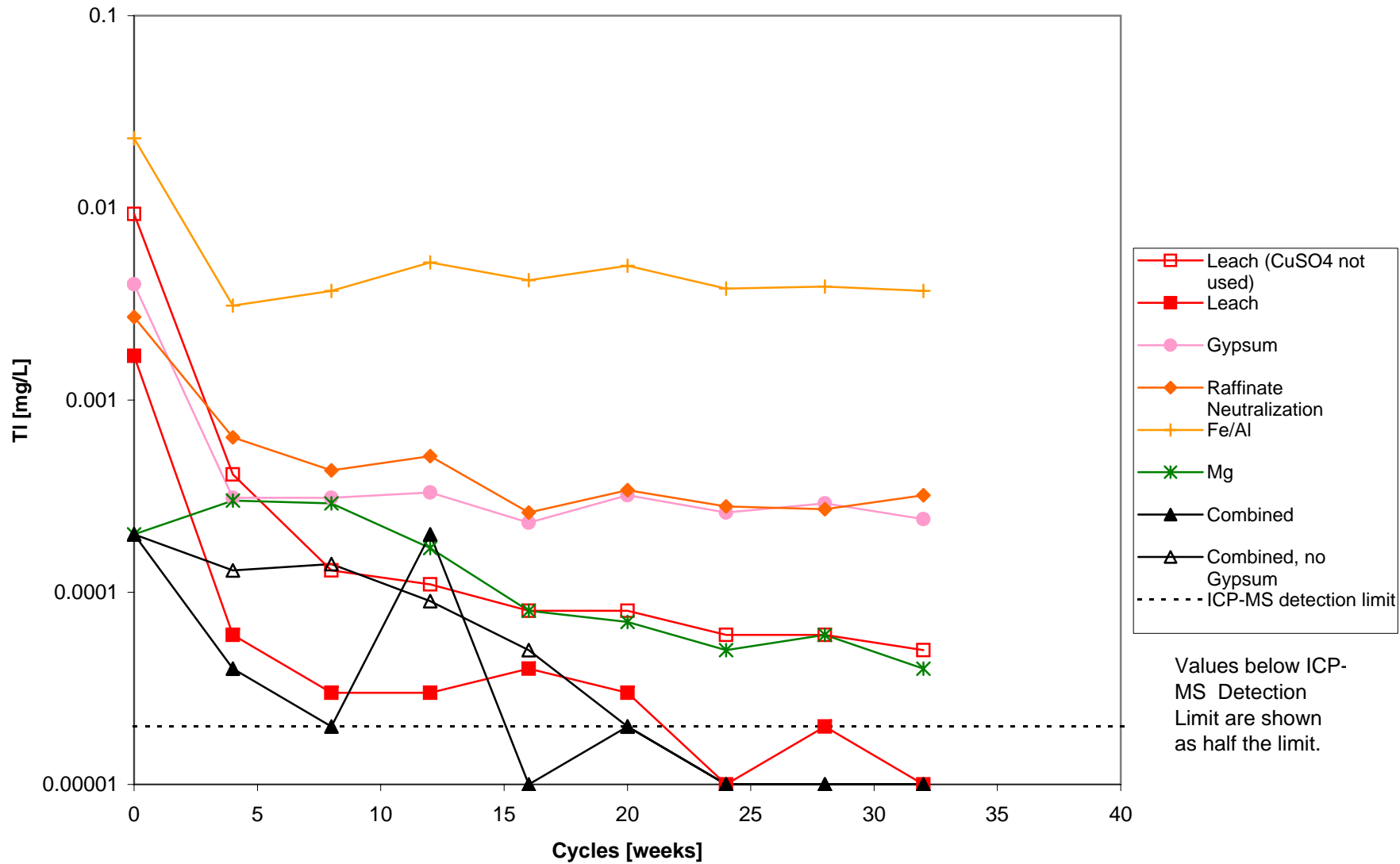


Humidity Cell - Concentrations
NorthMet Project

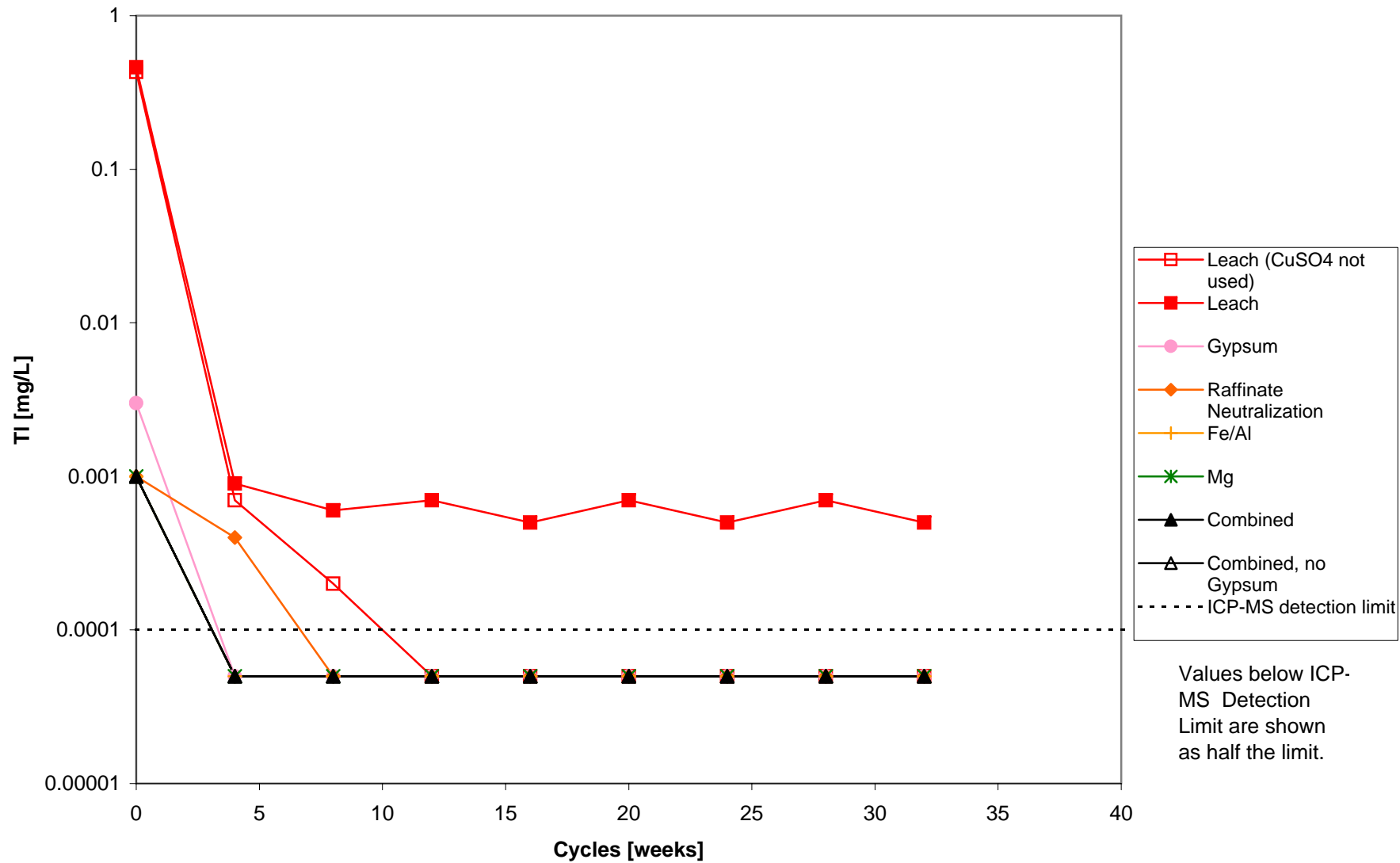




Humidity Cell - Concentrations
NorthMet Project

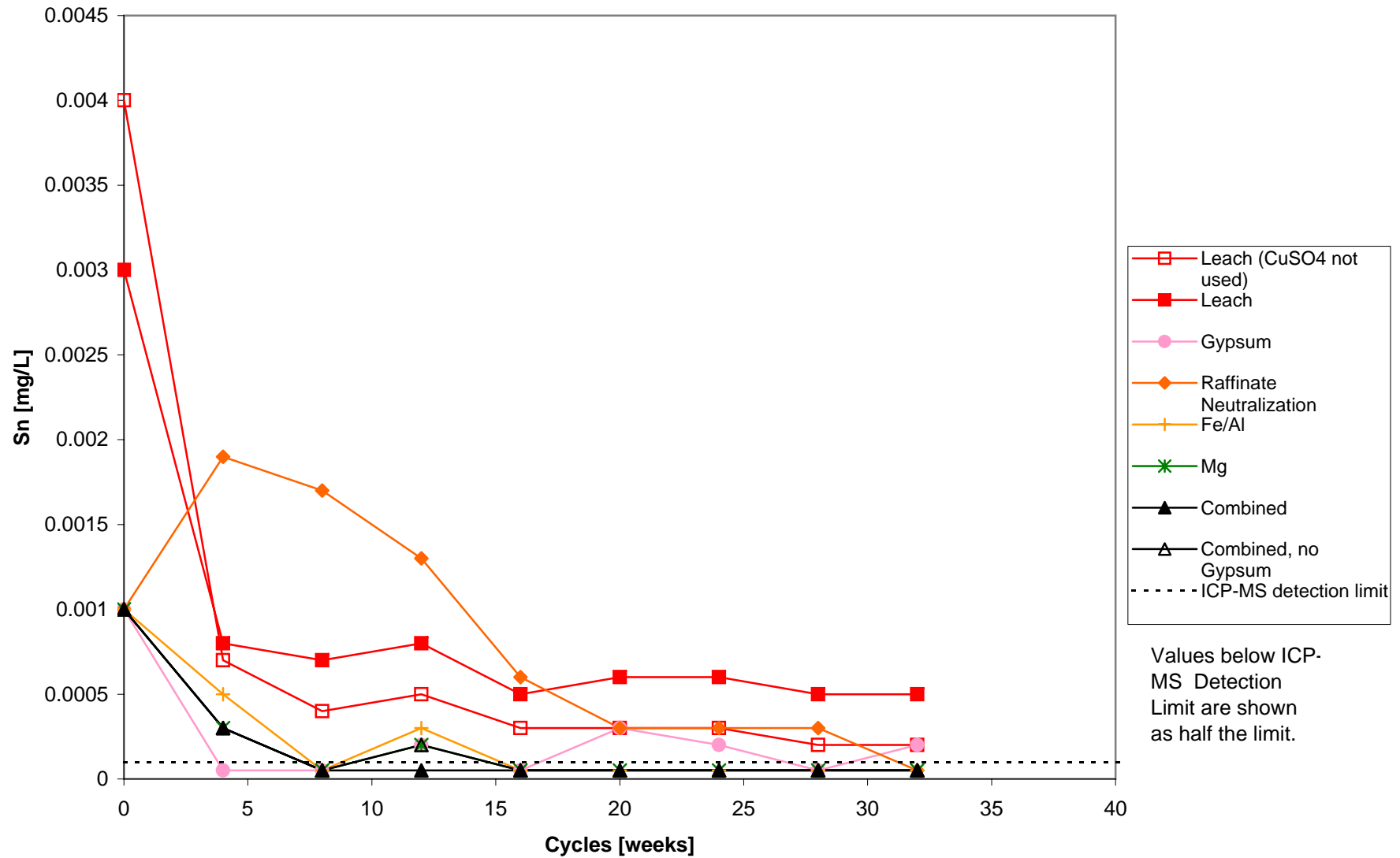


Humidity Cell - Concentrations
NorthMet Project

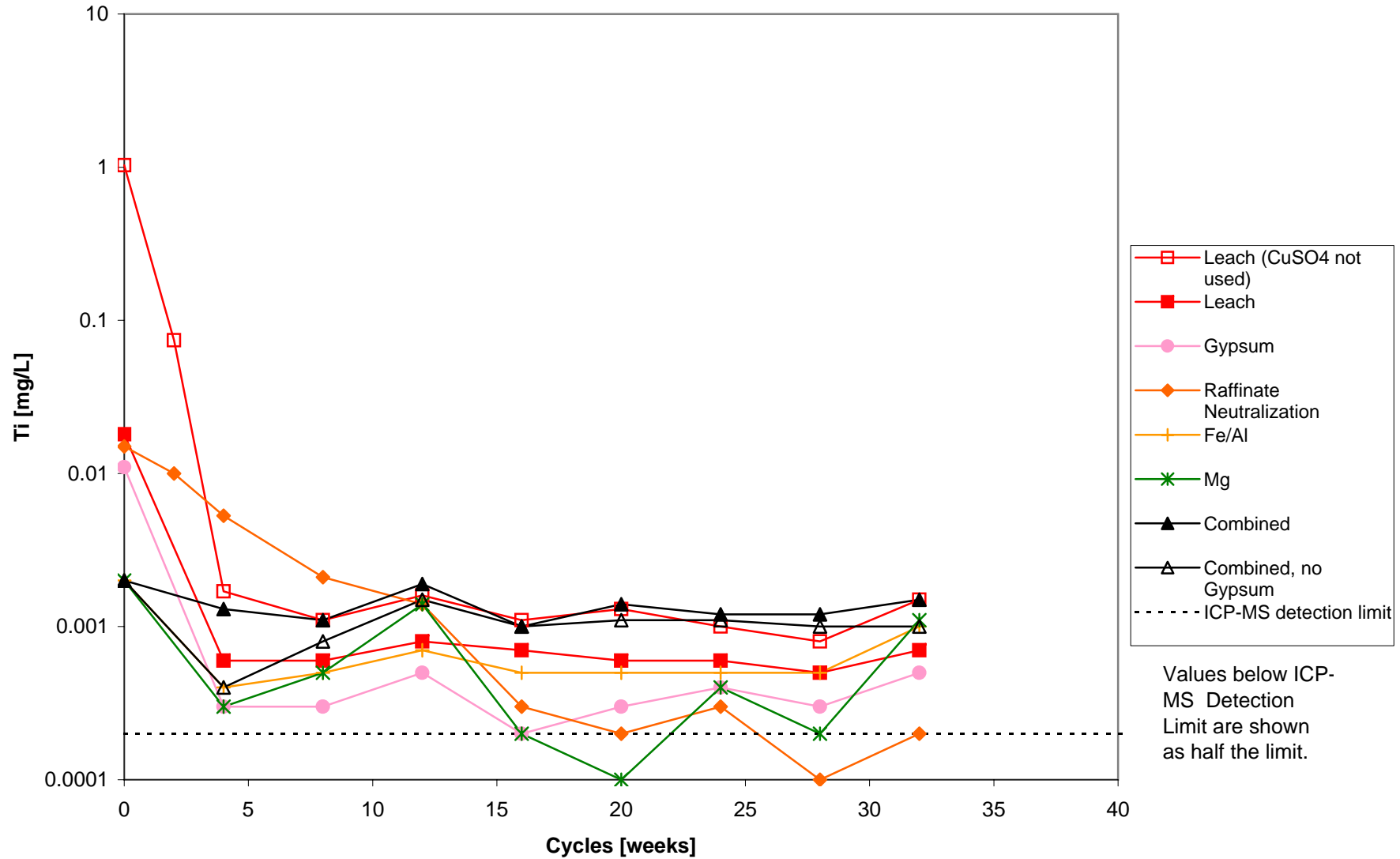


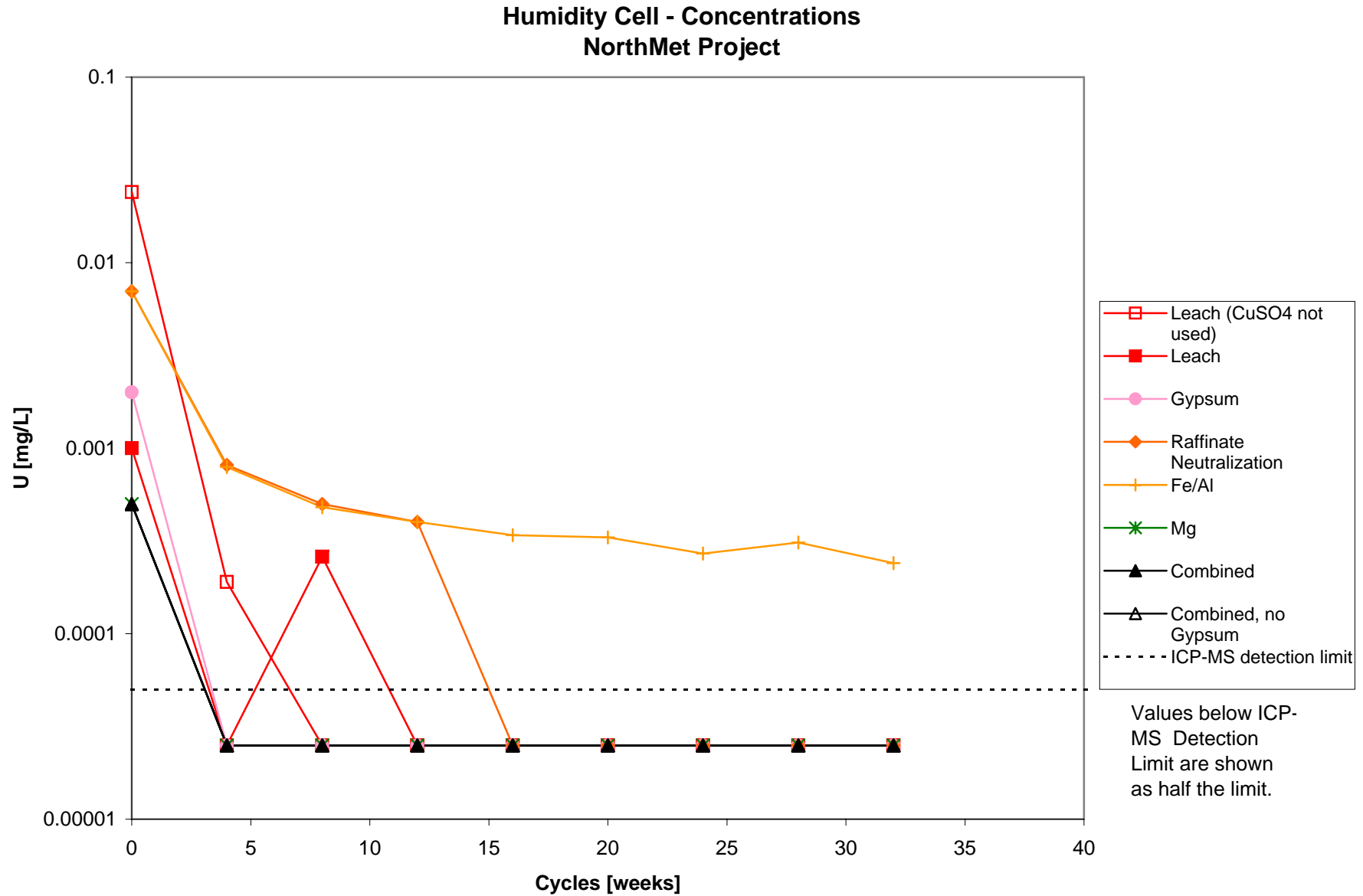
Values below ICP-MS Detection Limit are shown as half the limit.

Humidity Cell - Concentrations
NorthMet Project

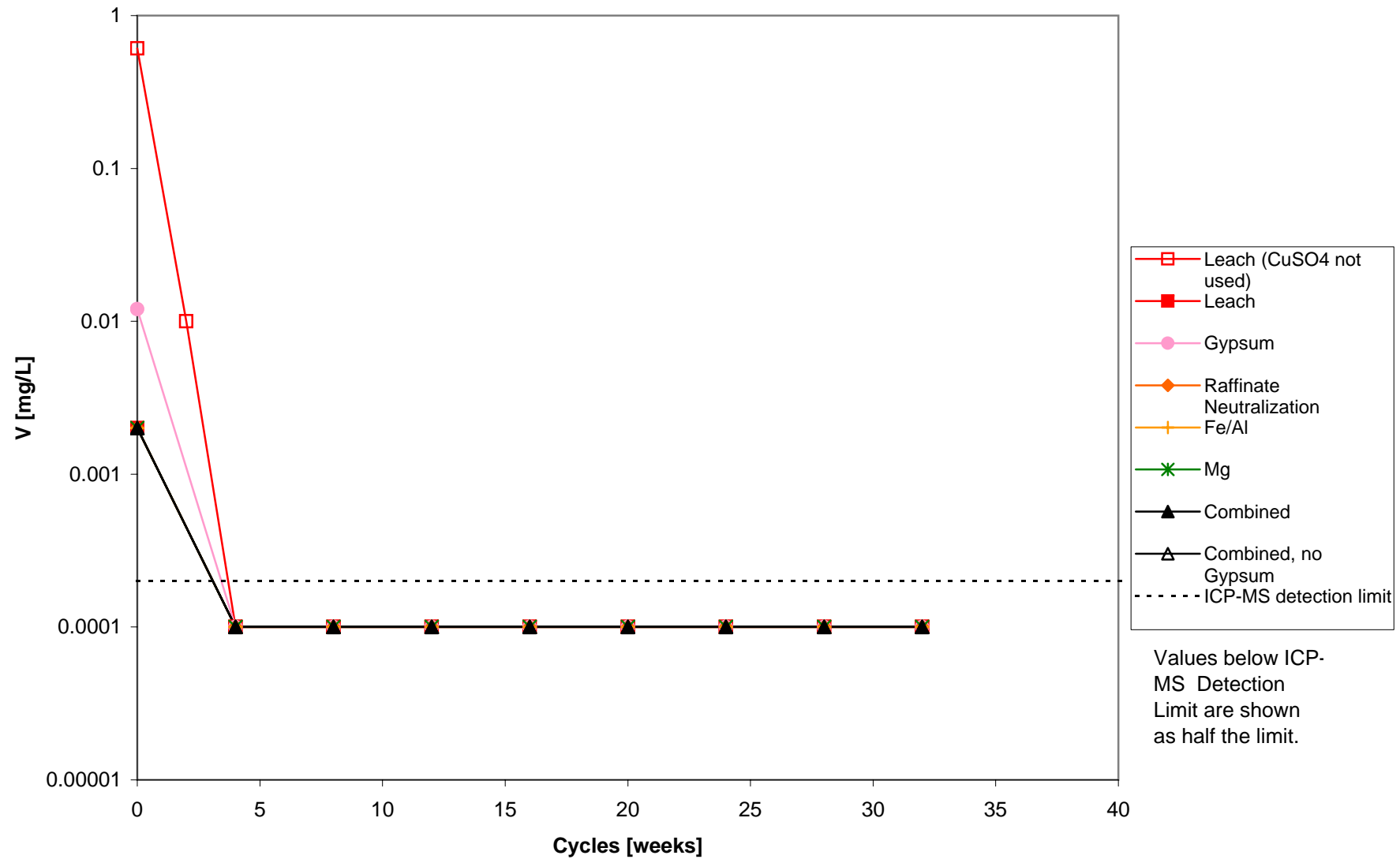


Humidity Cell - Concentrations
NorthMet Project

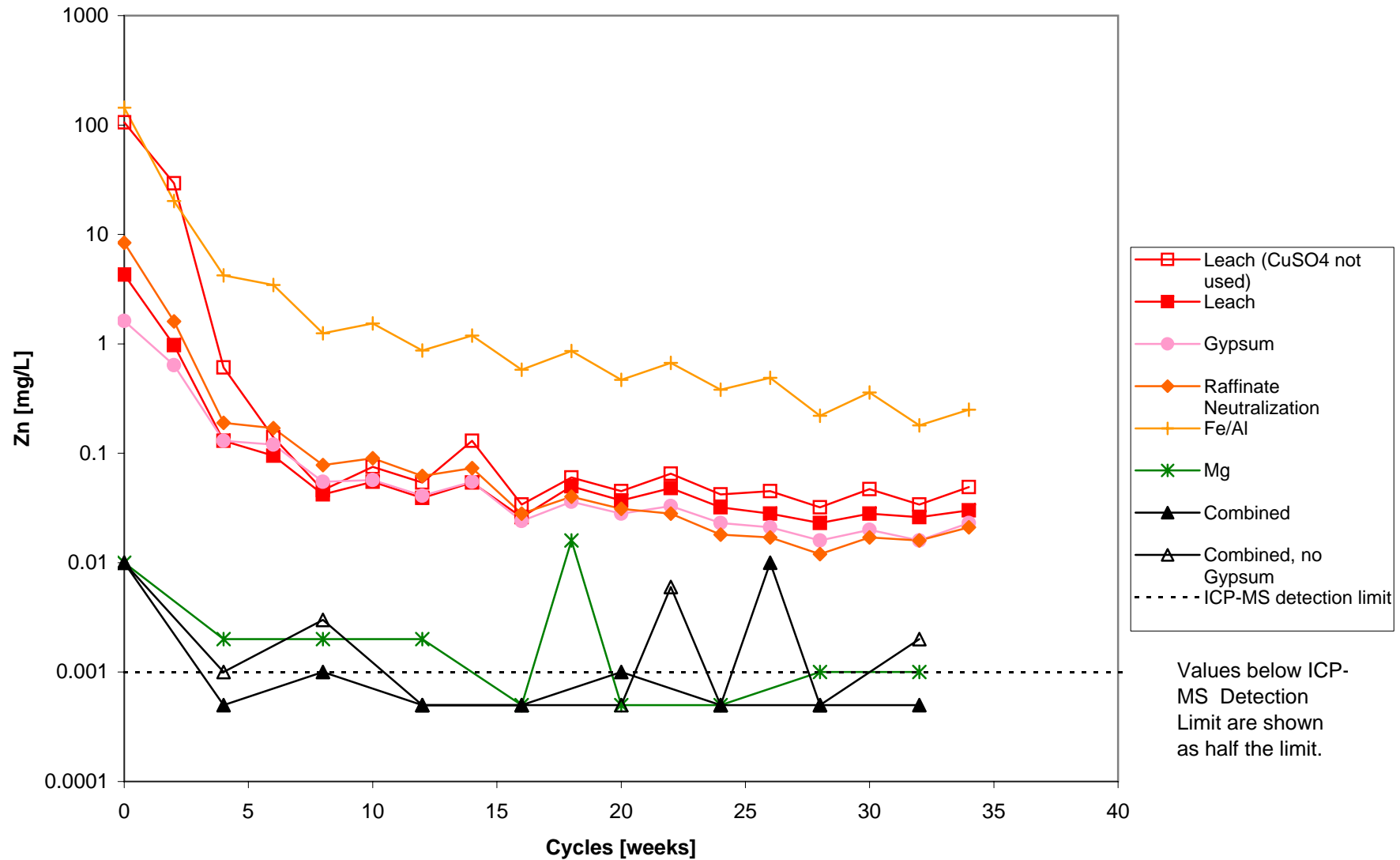




Humidity Cell - Concentrations
NorthMet Project

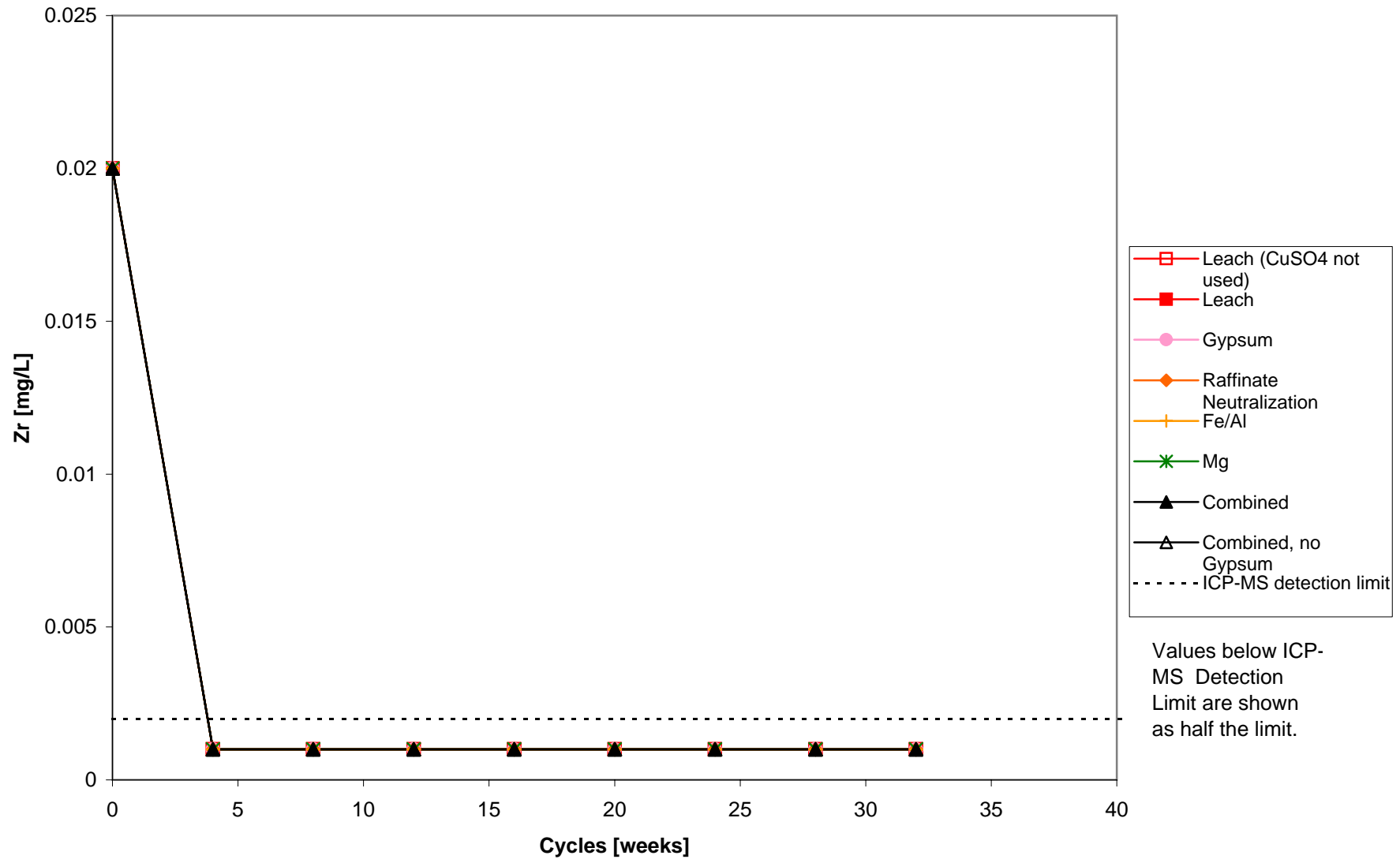


Humidity Cell - Concentrations
NorthMet Project

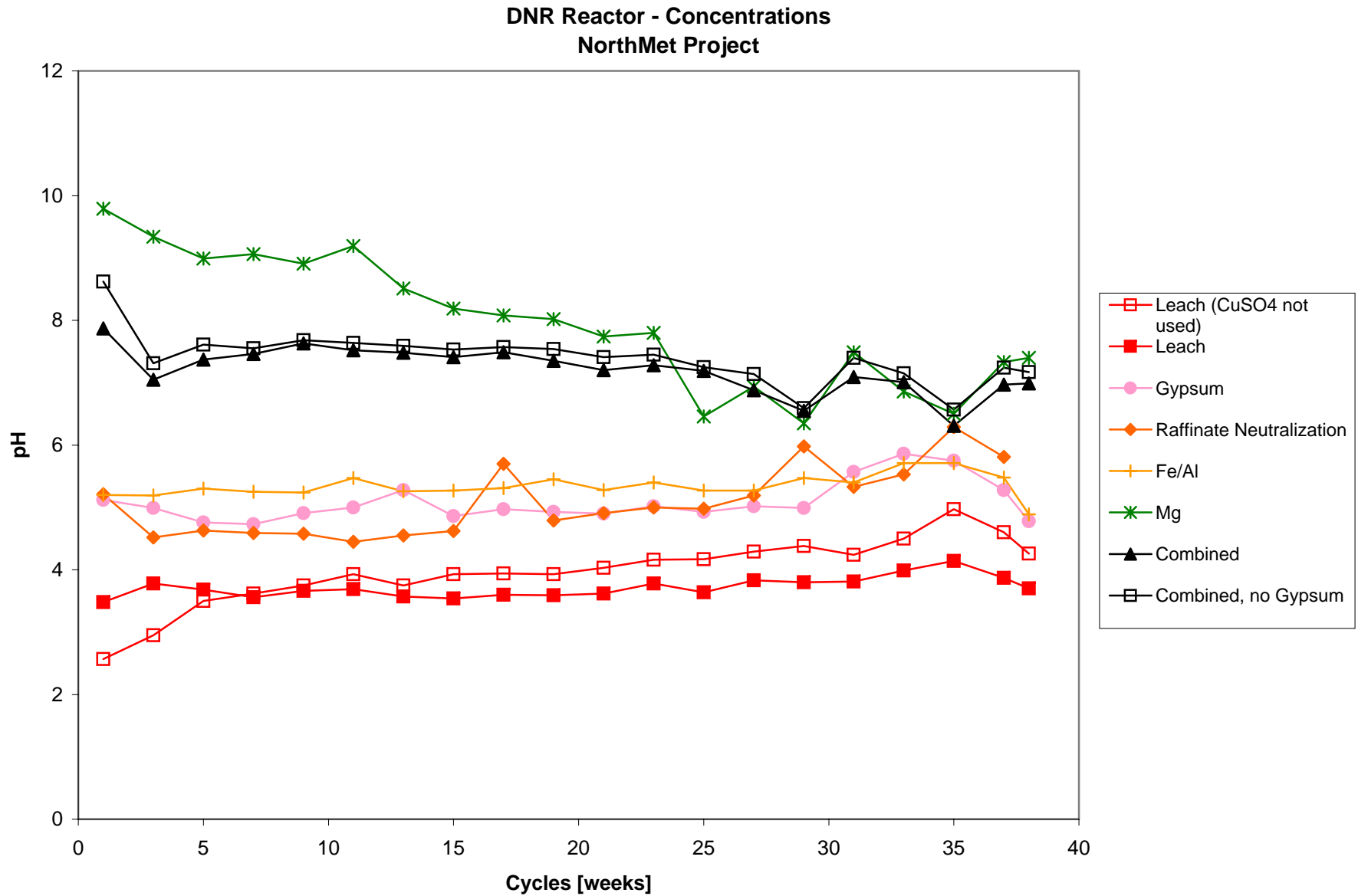


Values below ICP-MS Detection Limit are shown as half the limit.

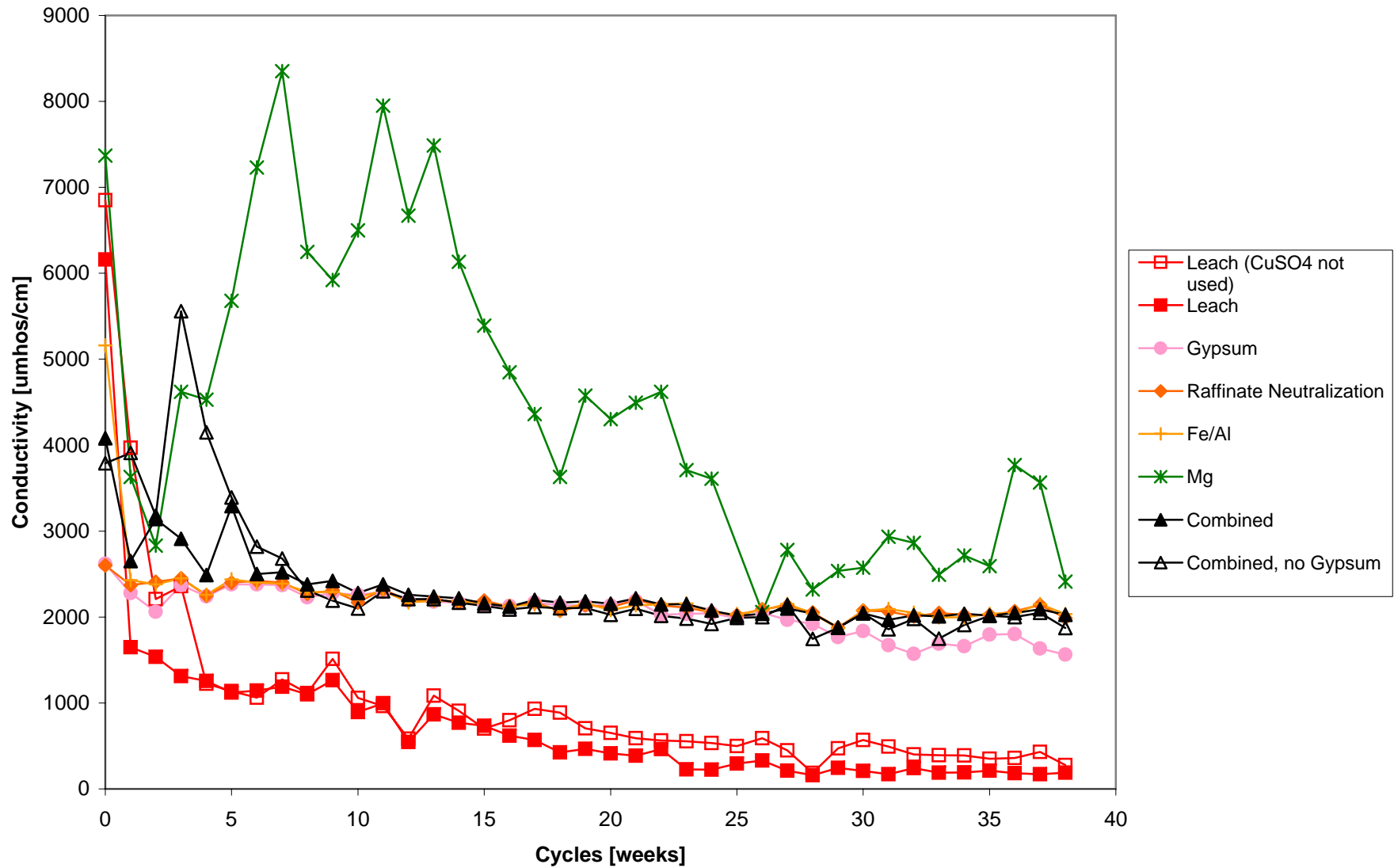
Humidity Cell - Concentrations
NorthMet Project

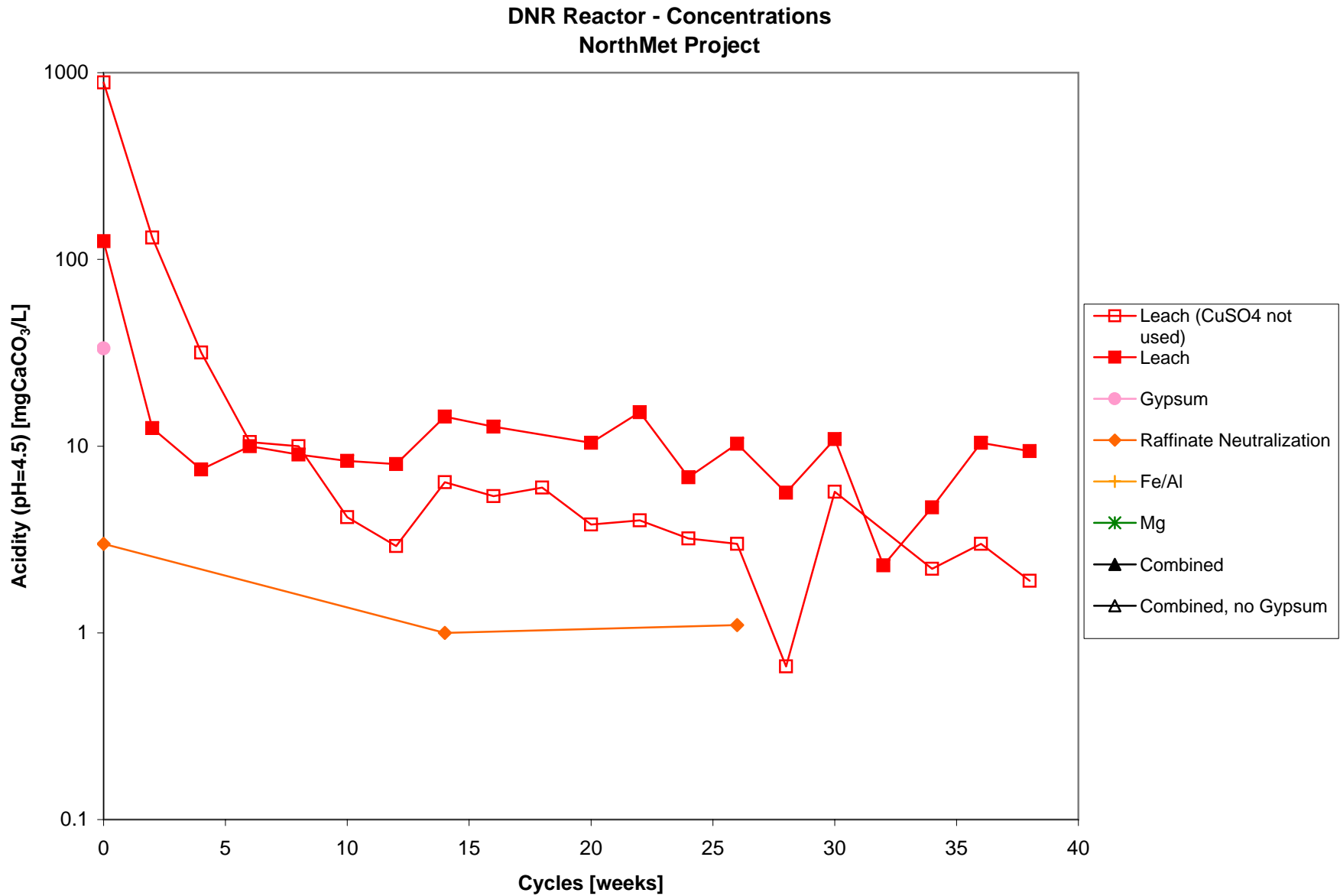


Appendix C.3
DNR Reactors

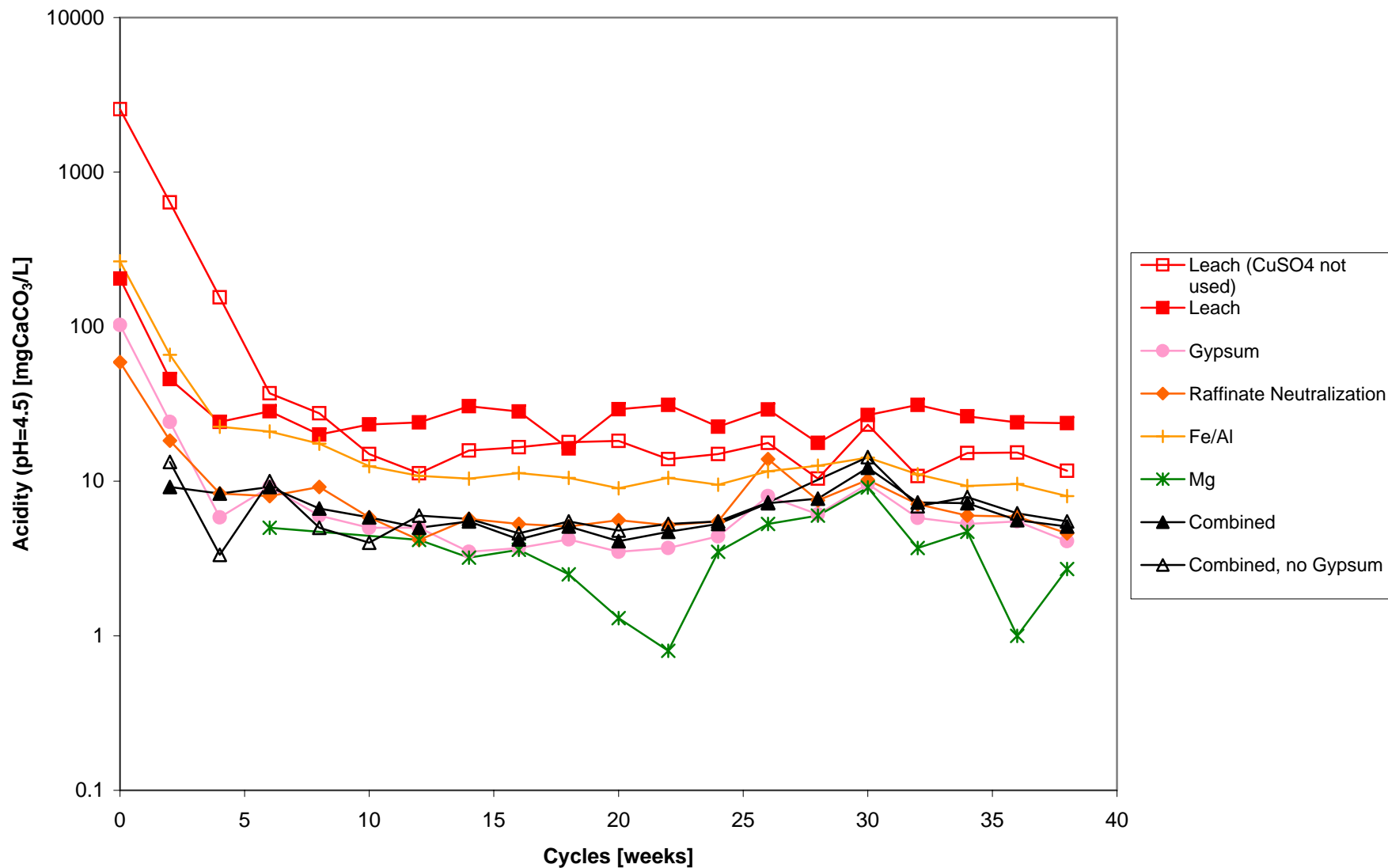


DNR Reactor - Concentrations
NorthMet Project

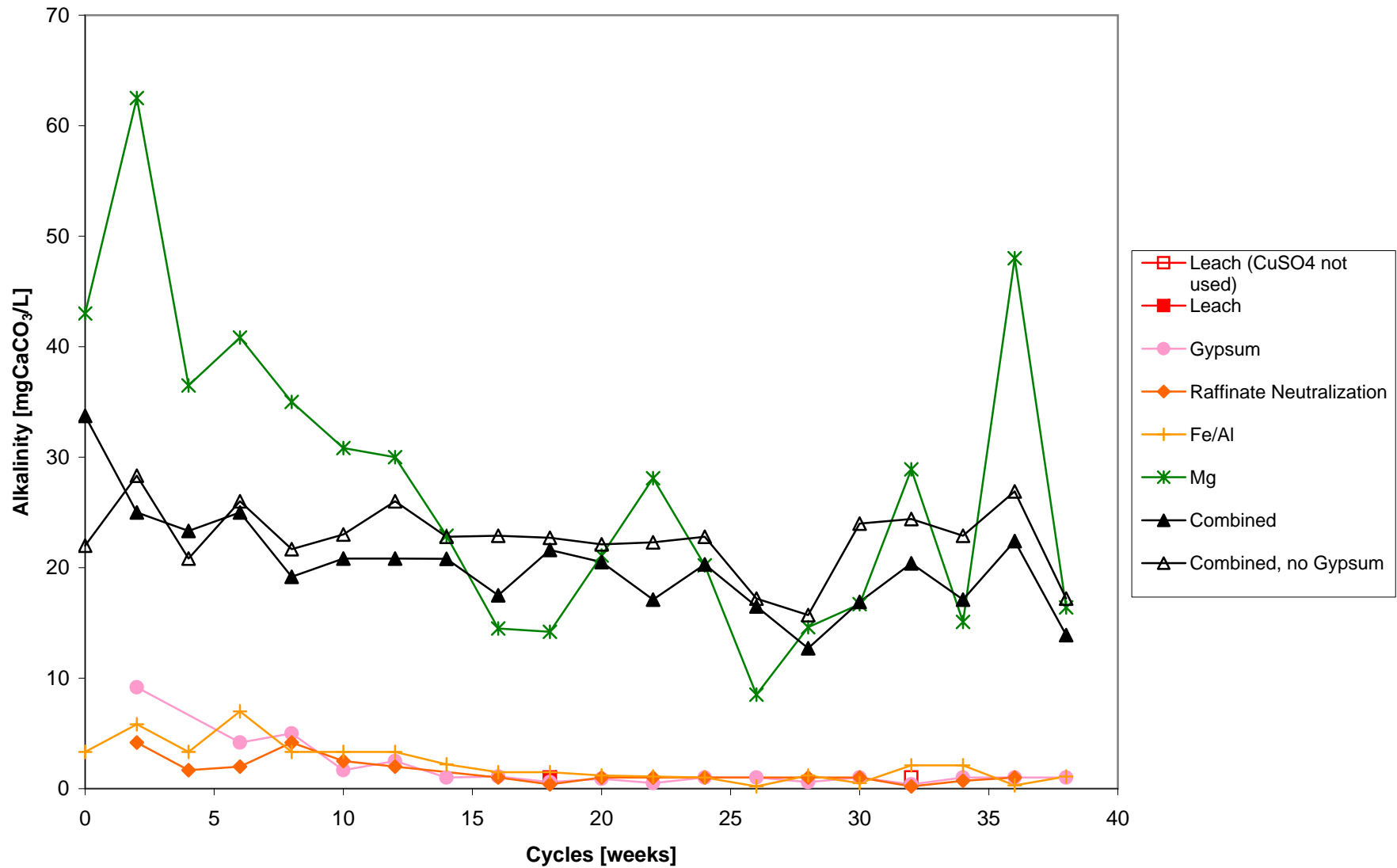




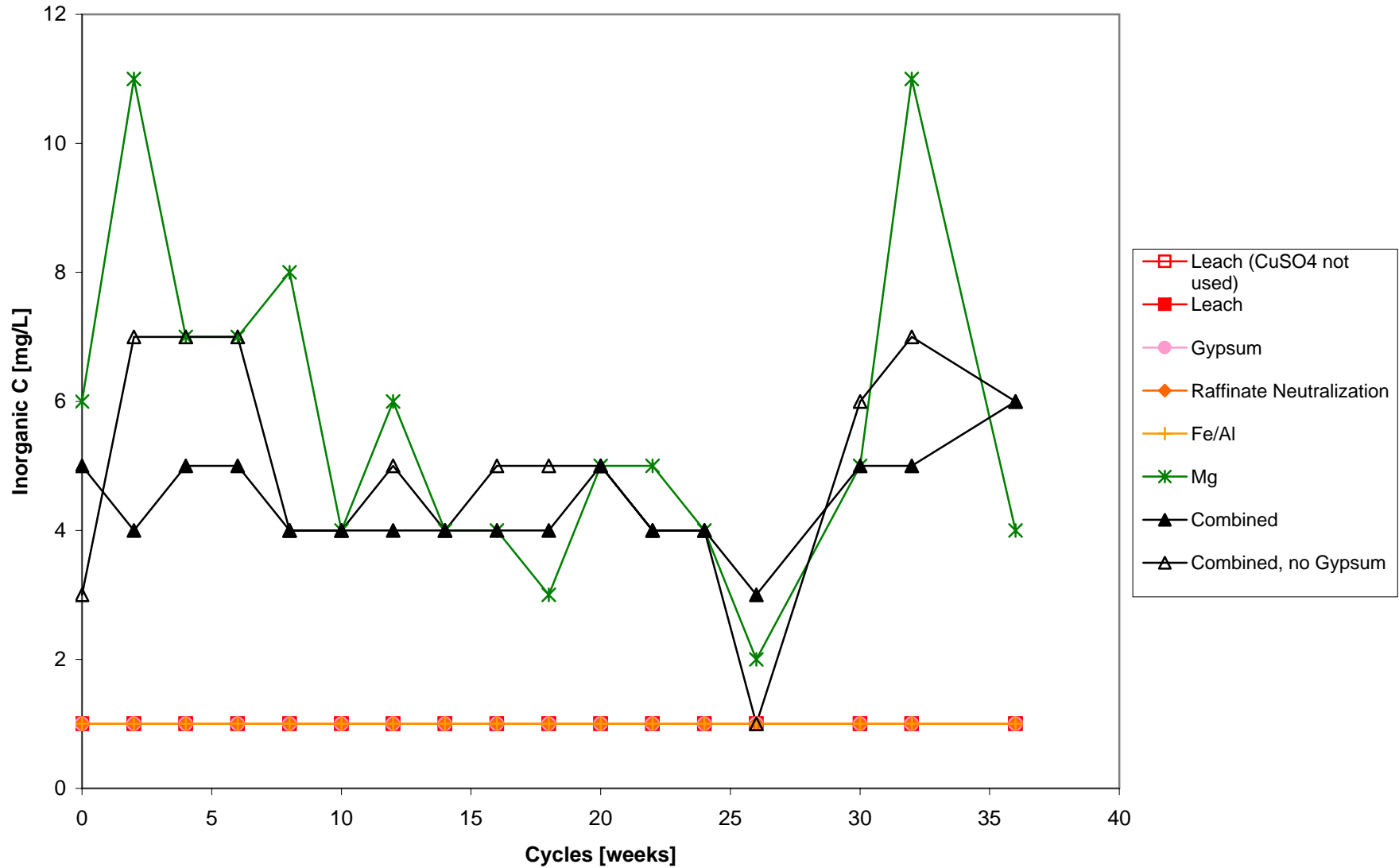
DNR Reactor - Concentrations
NorthMet Project

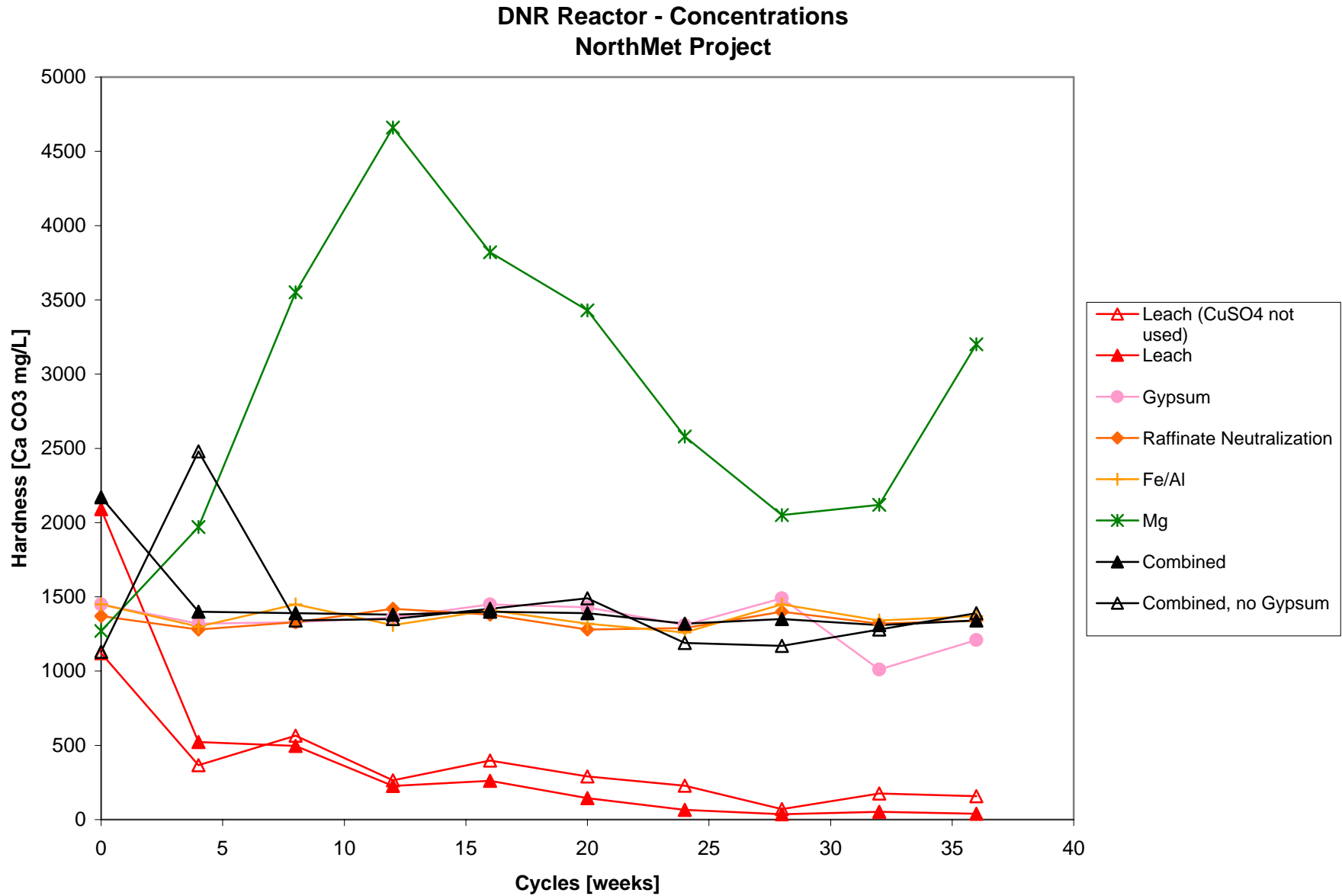


DNR Reactor - Concentrations
NorthMet Project

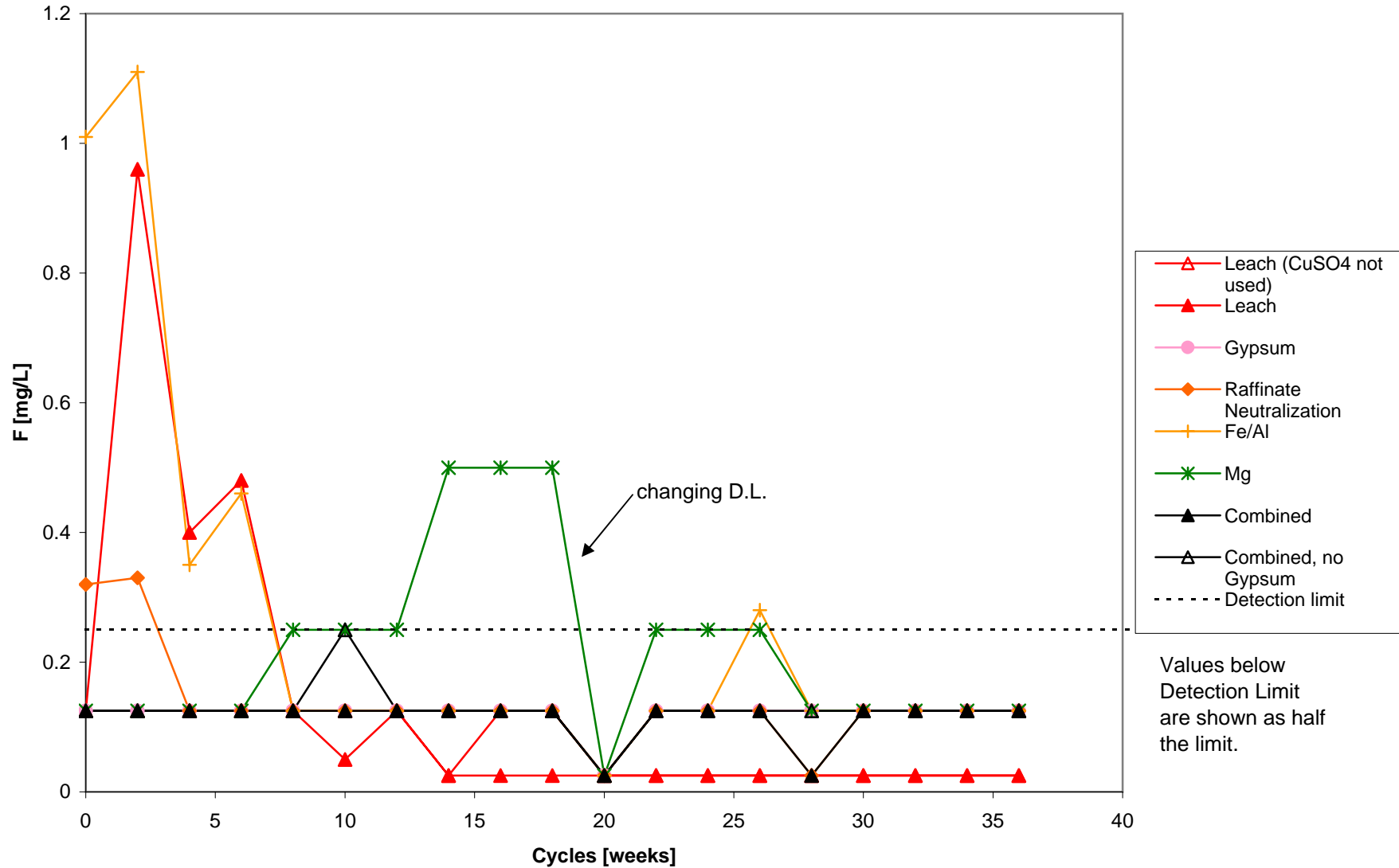


DNR Reactor - Concentrations
NorthMet Project

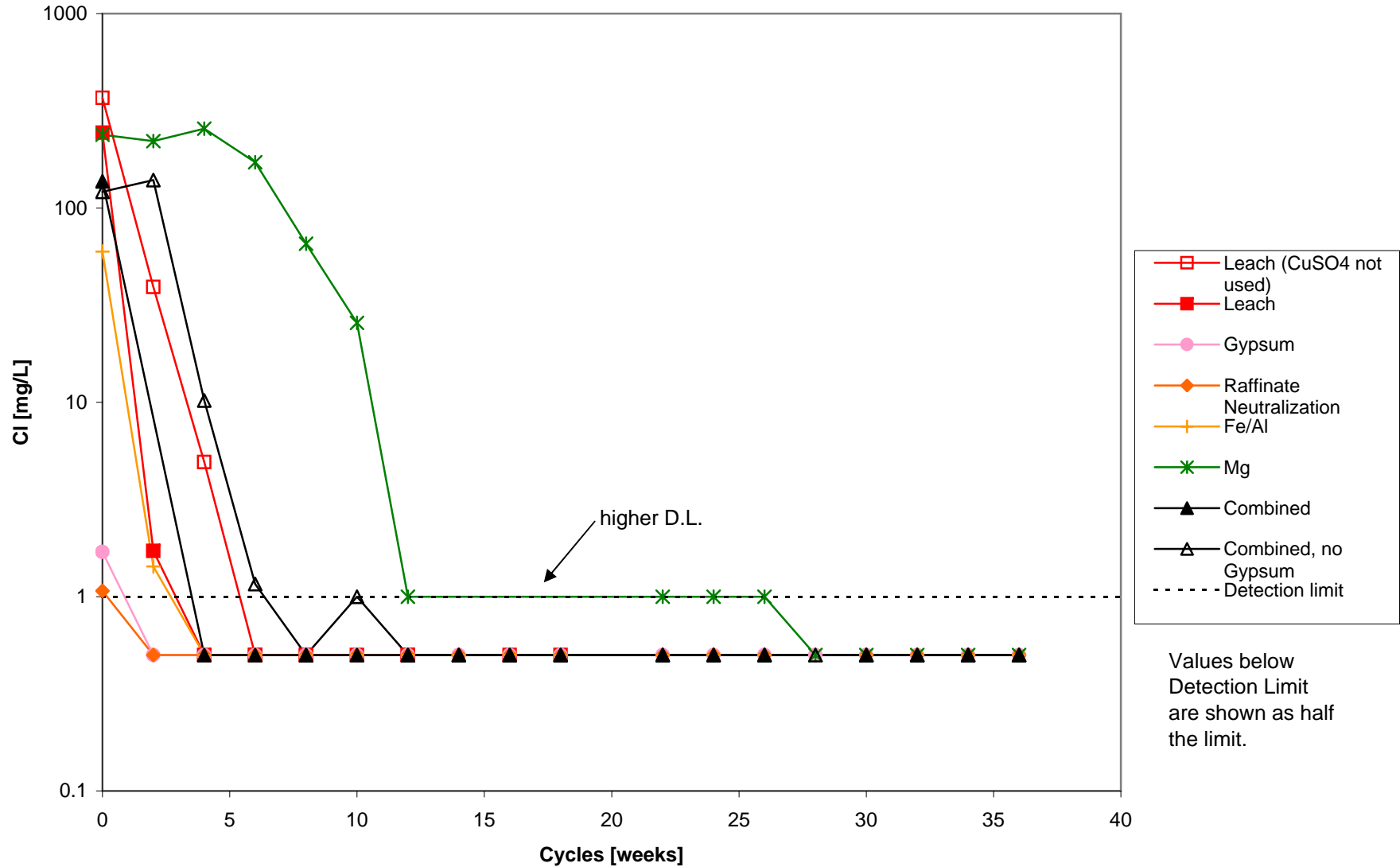




DNR Reactor - Concentrations
NorthMet Project

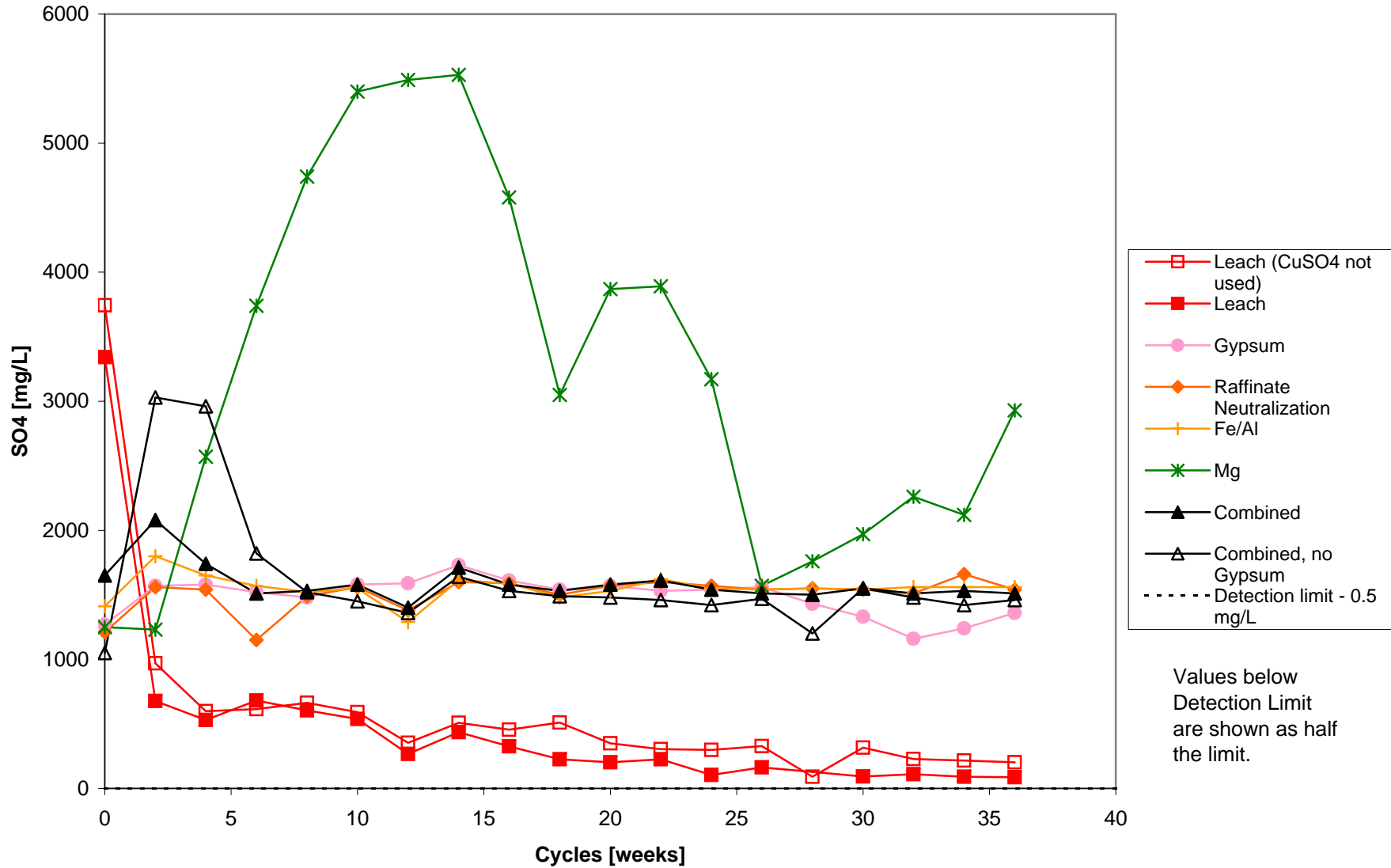


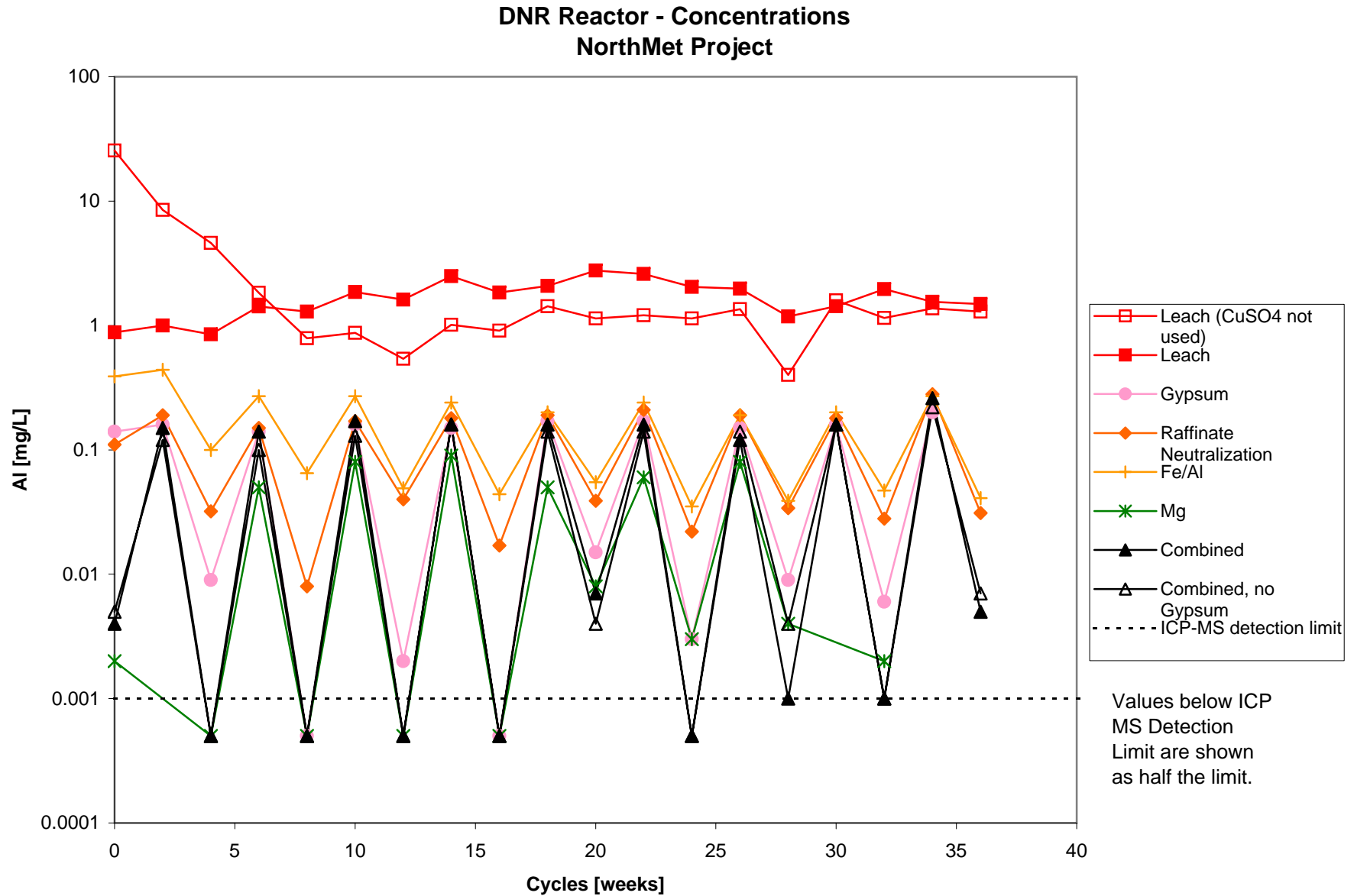
DNR Reactor - Concentrations
NorthMet Project



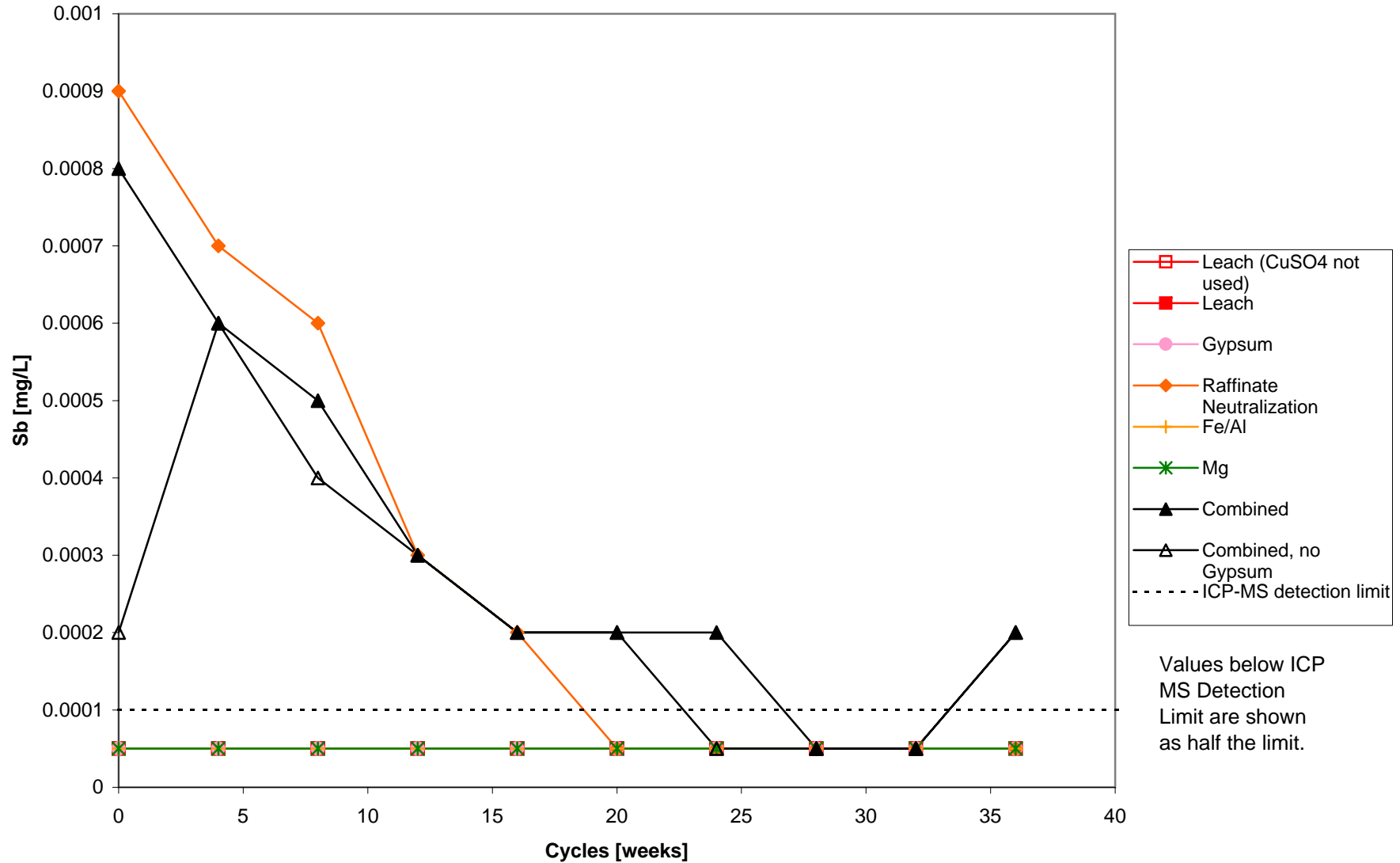
Values below
Detection Limit
are shown as half
the limit.

DNR Reactor - Concentrations
NorthMet Project

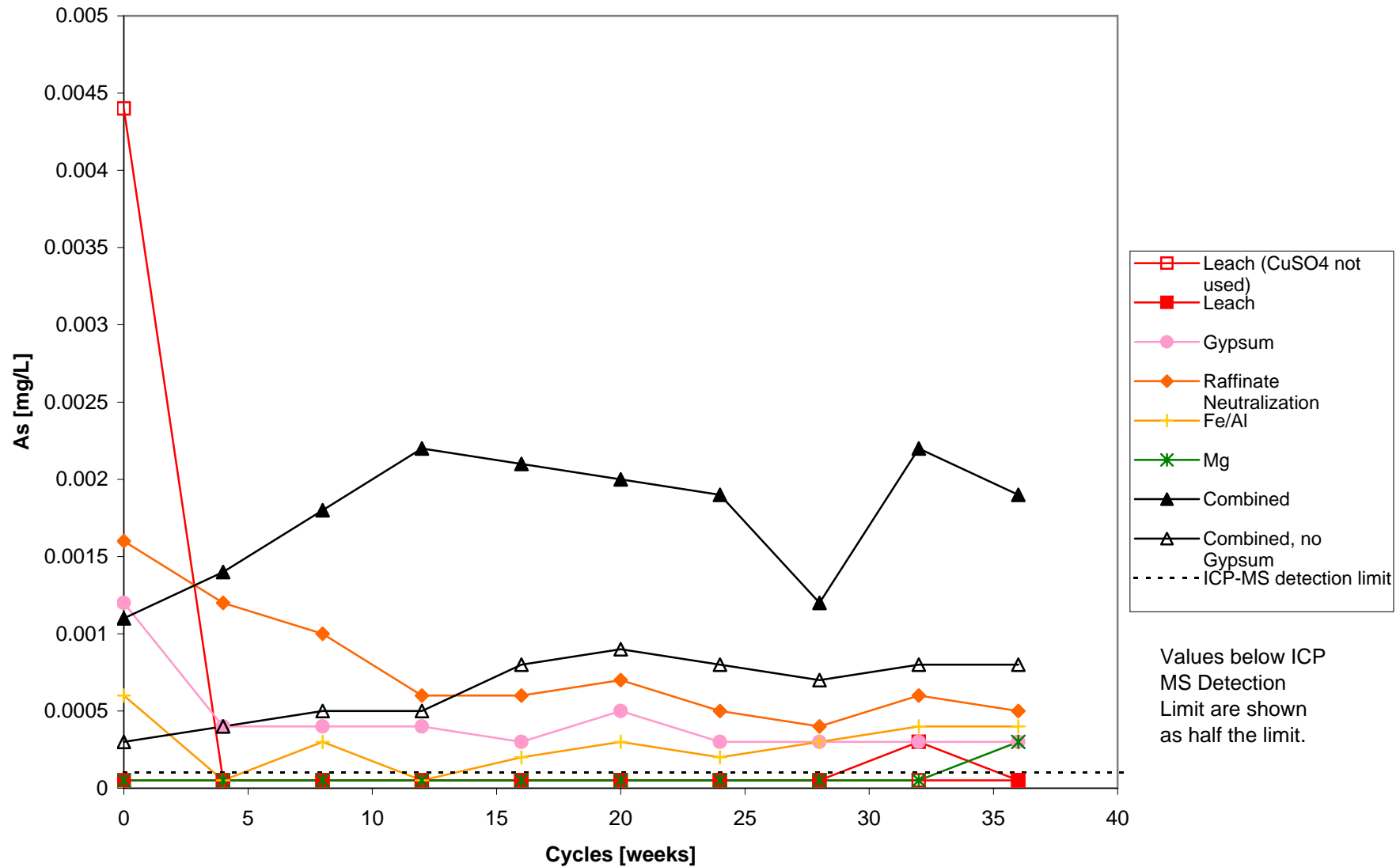


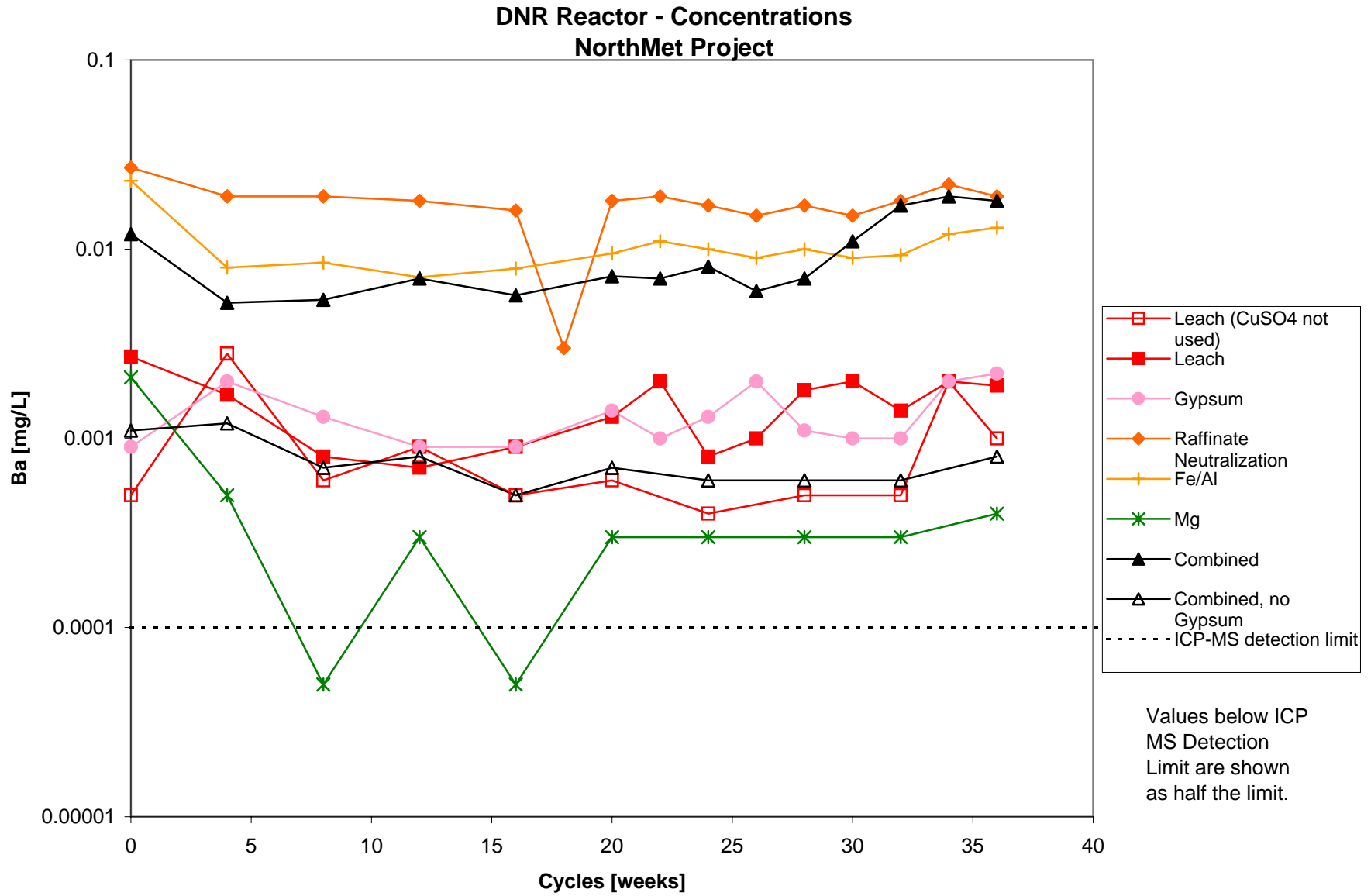


DNR Reactor - Concentrations
NorthMet Project

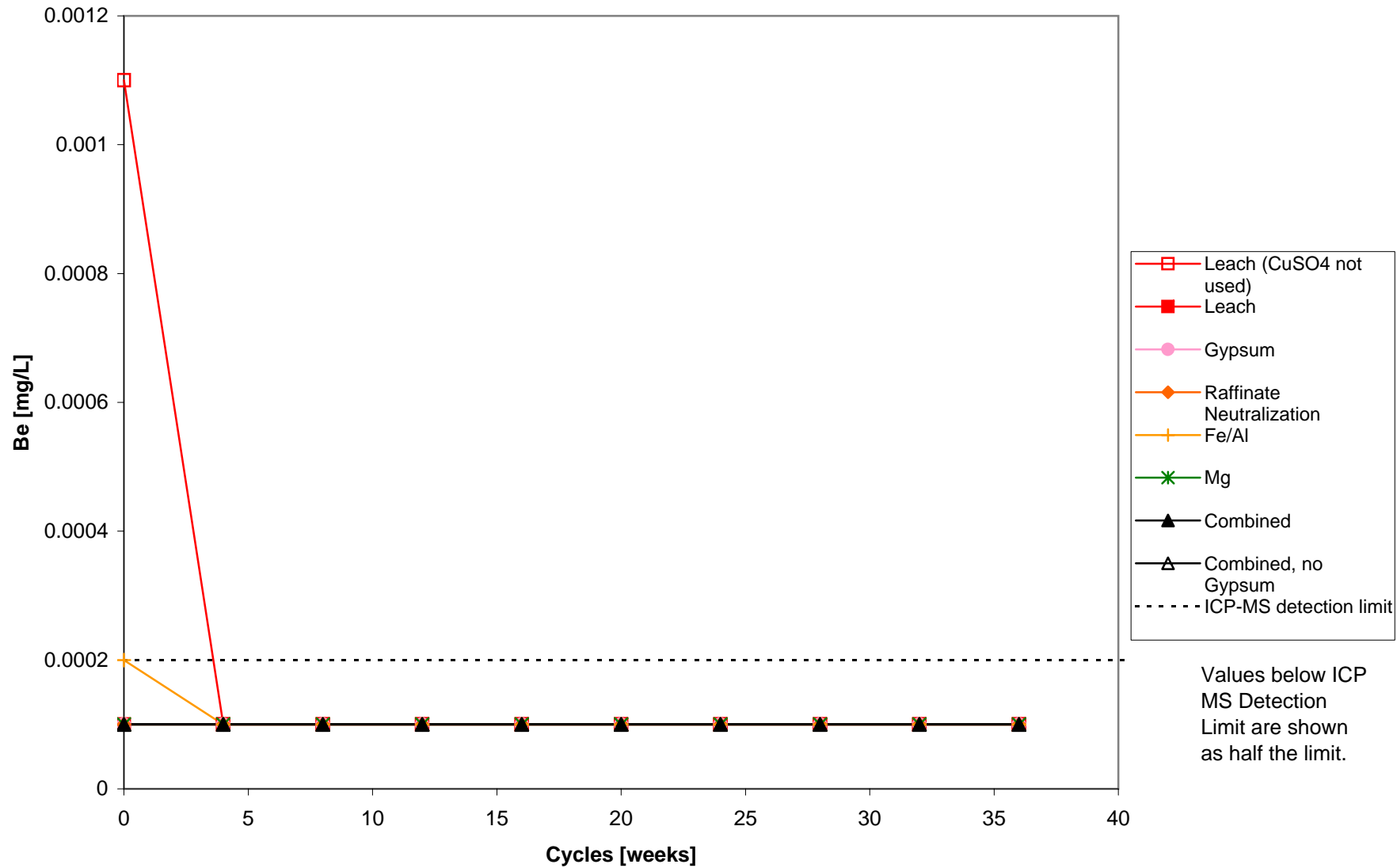


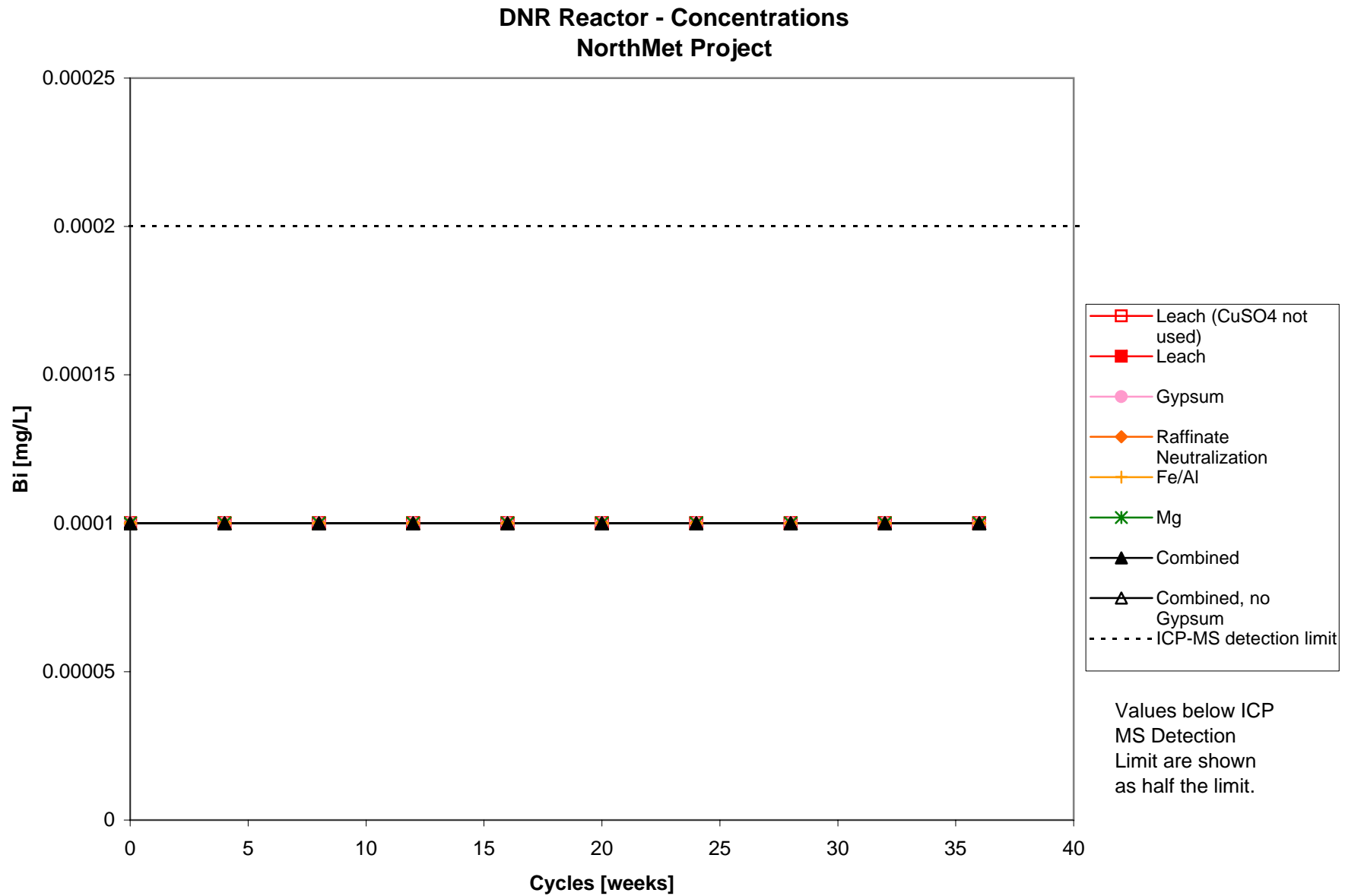
DNR Reactor - Concentrations
NorthMet Project

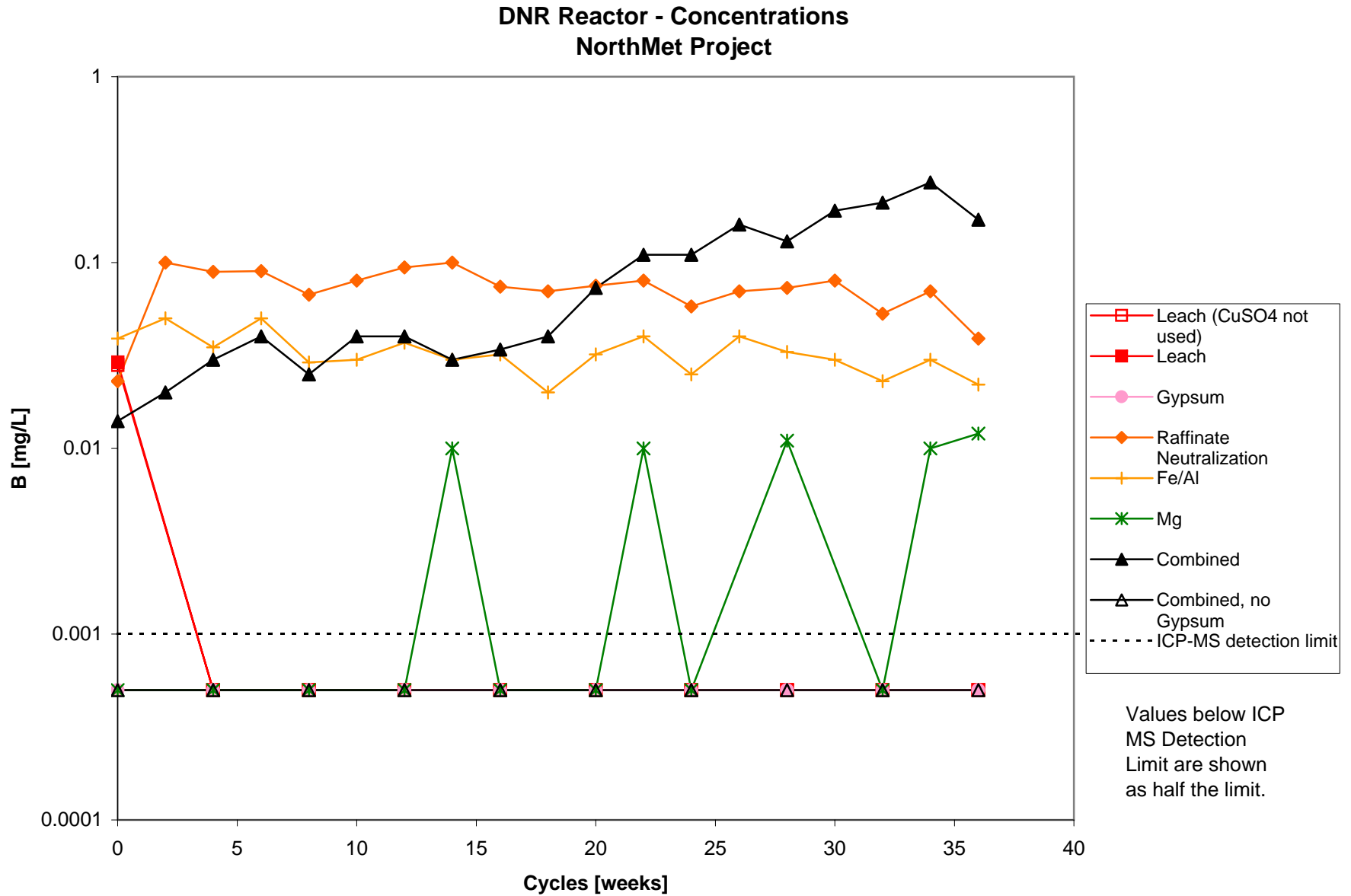




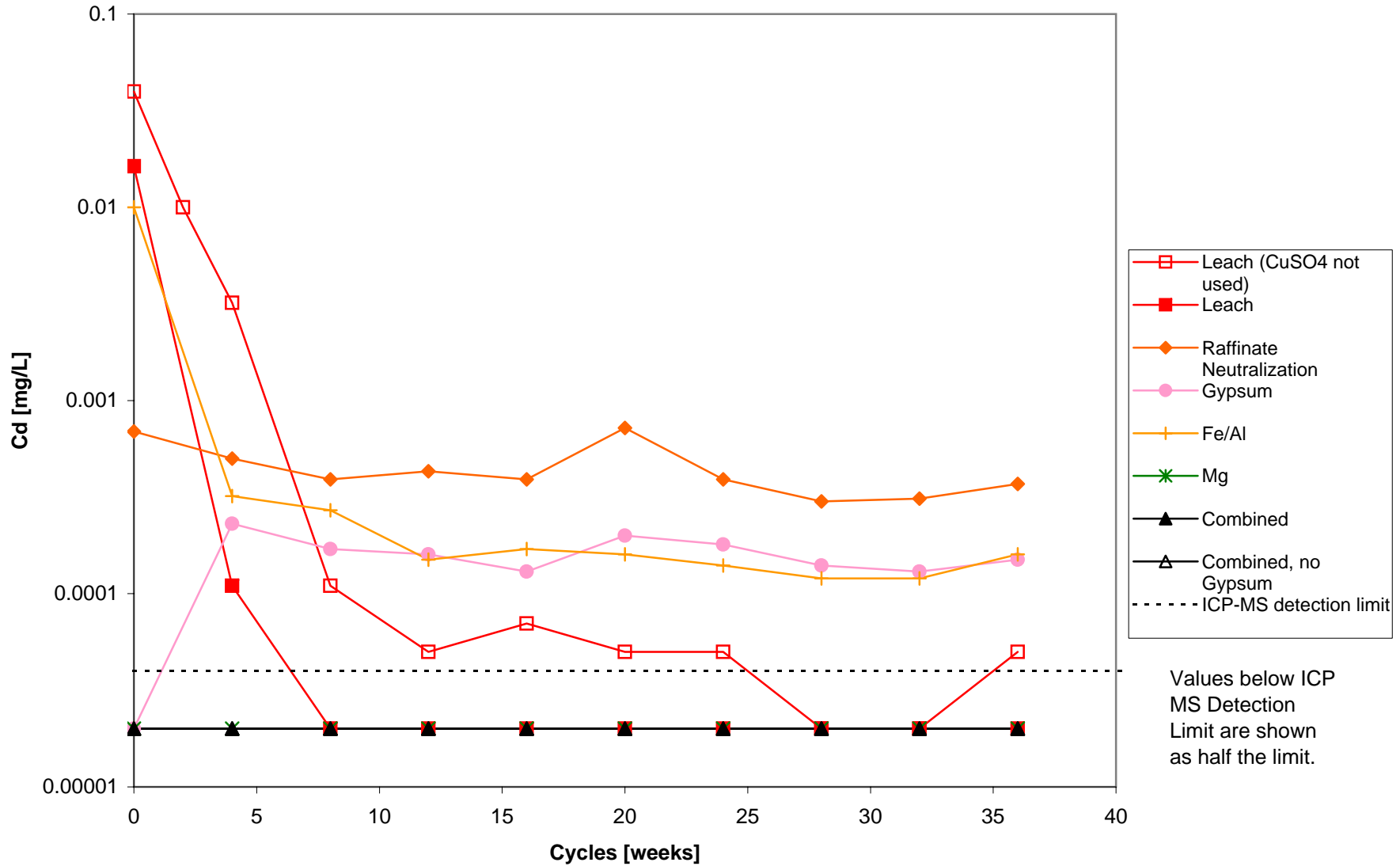
DNR Reactor - Concentrations
NorthMet Project





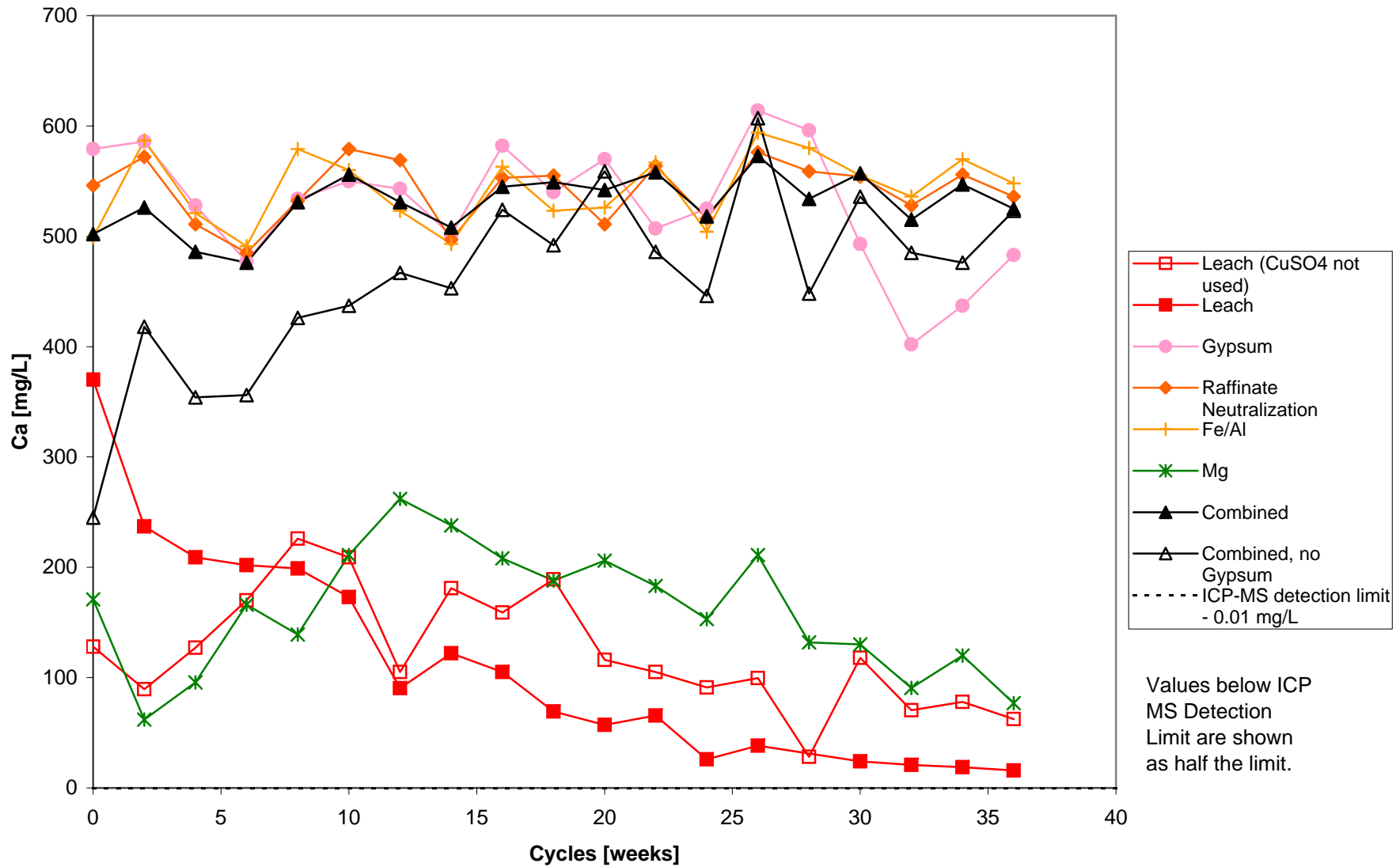


DNR Reactor - Concentrations
NorthMet Project

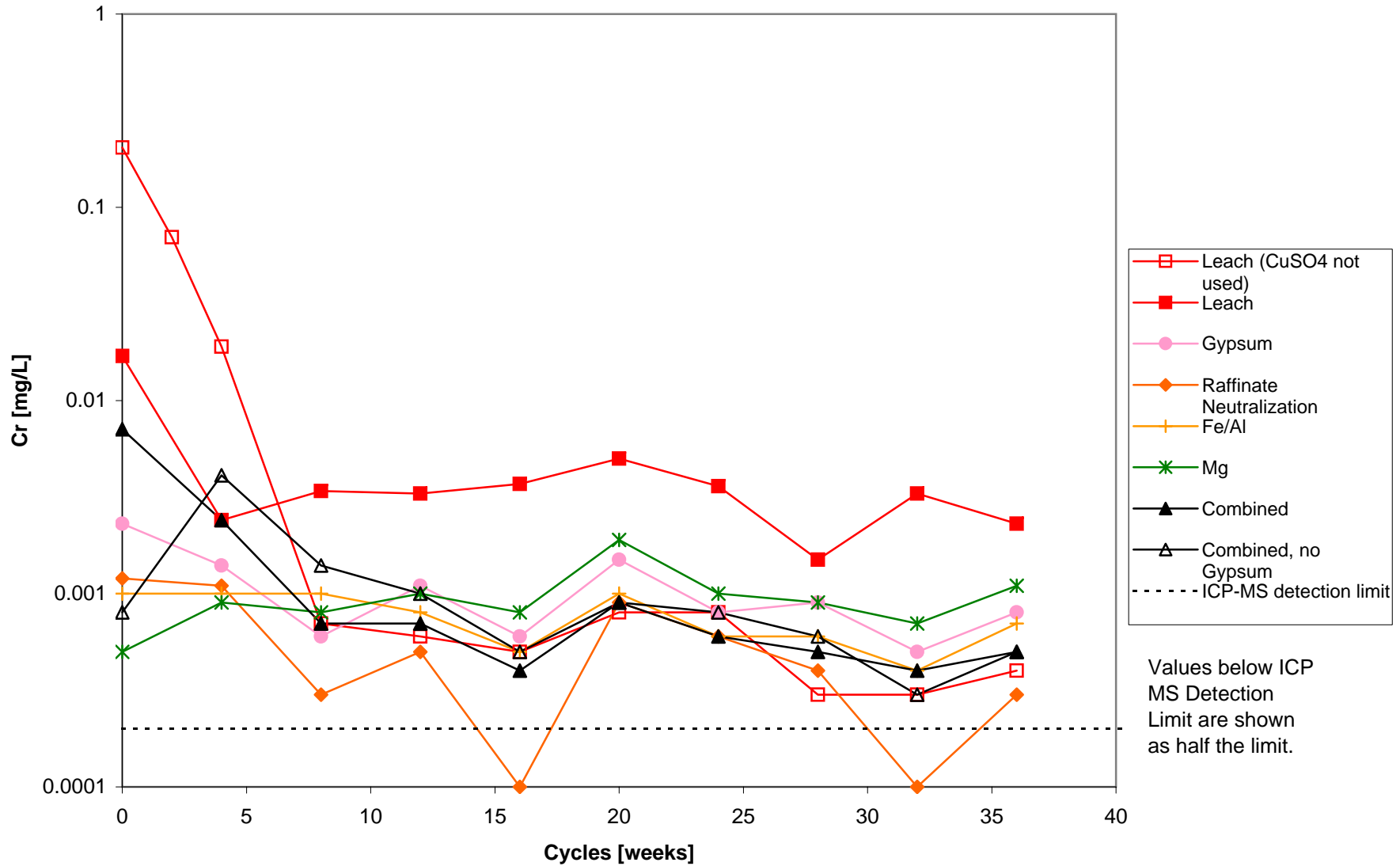


Values below ICP
MS Detection
Limit are shown
as half the limit.

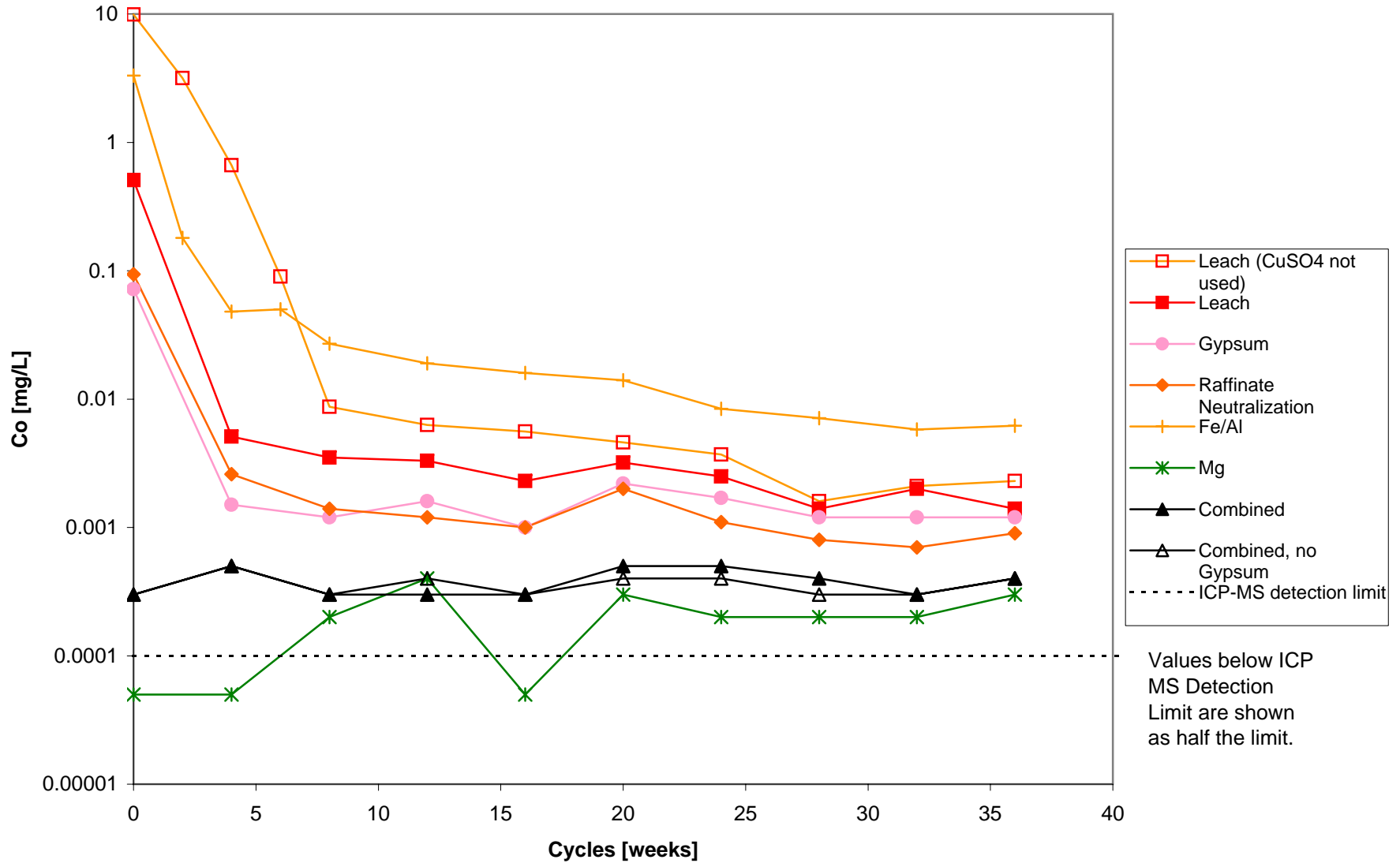
DNR Reactor - Concentrations
NorthMet Project

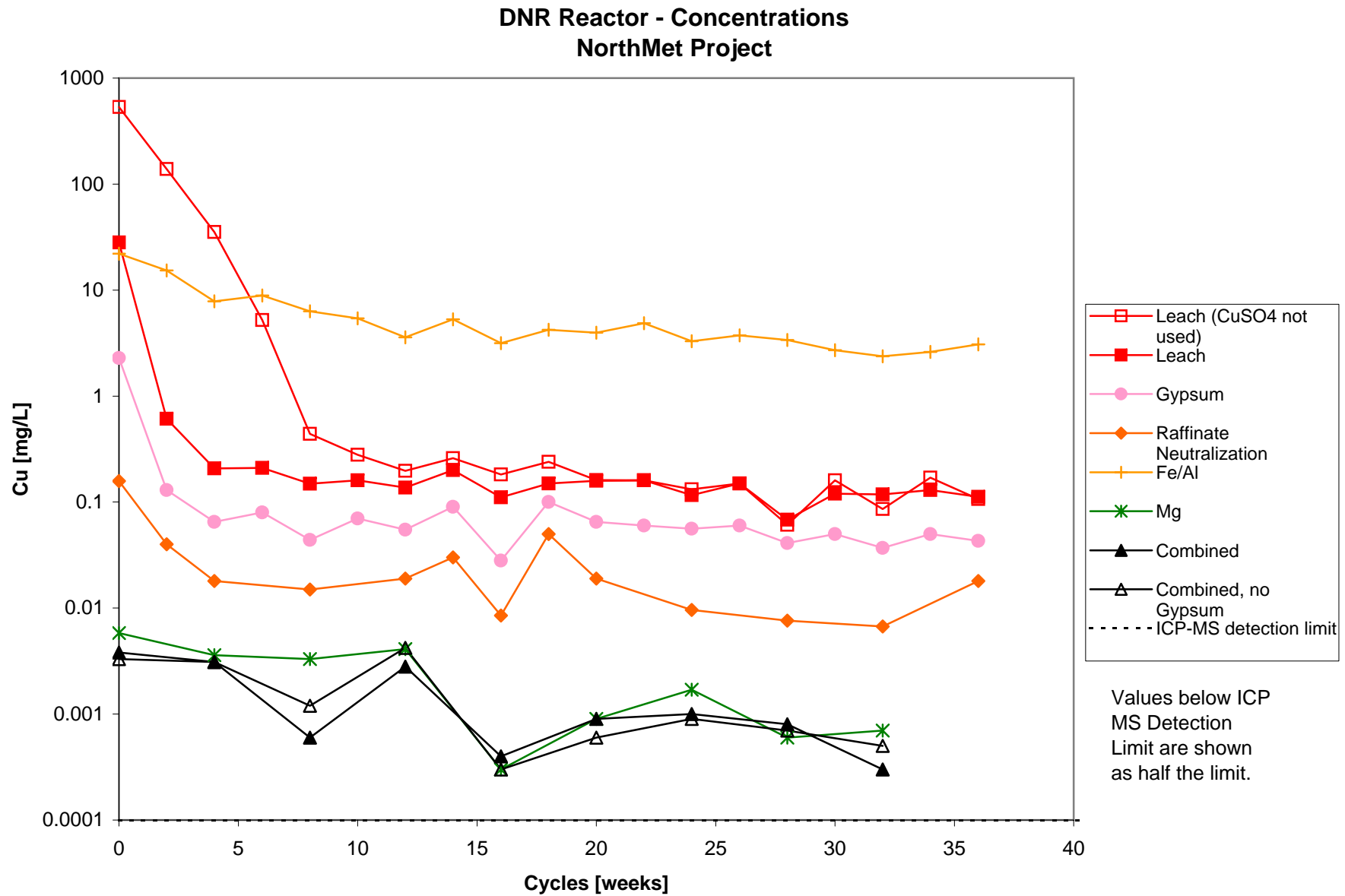


DNR Reactor - Concentrations
NorthMet Project

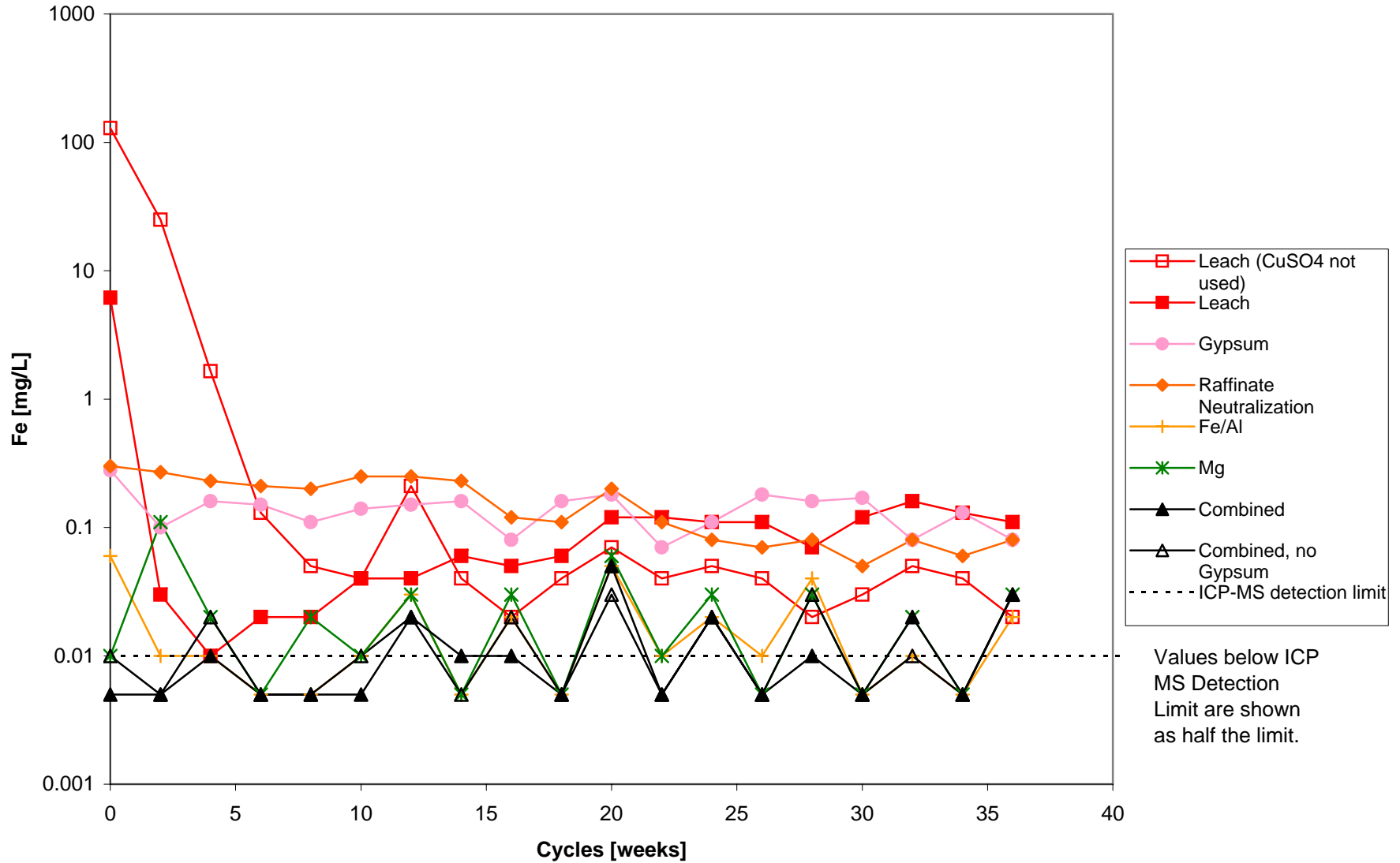


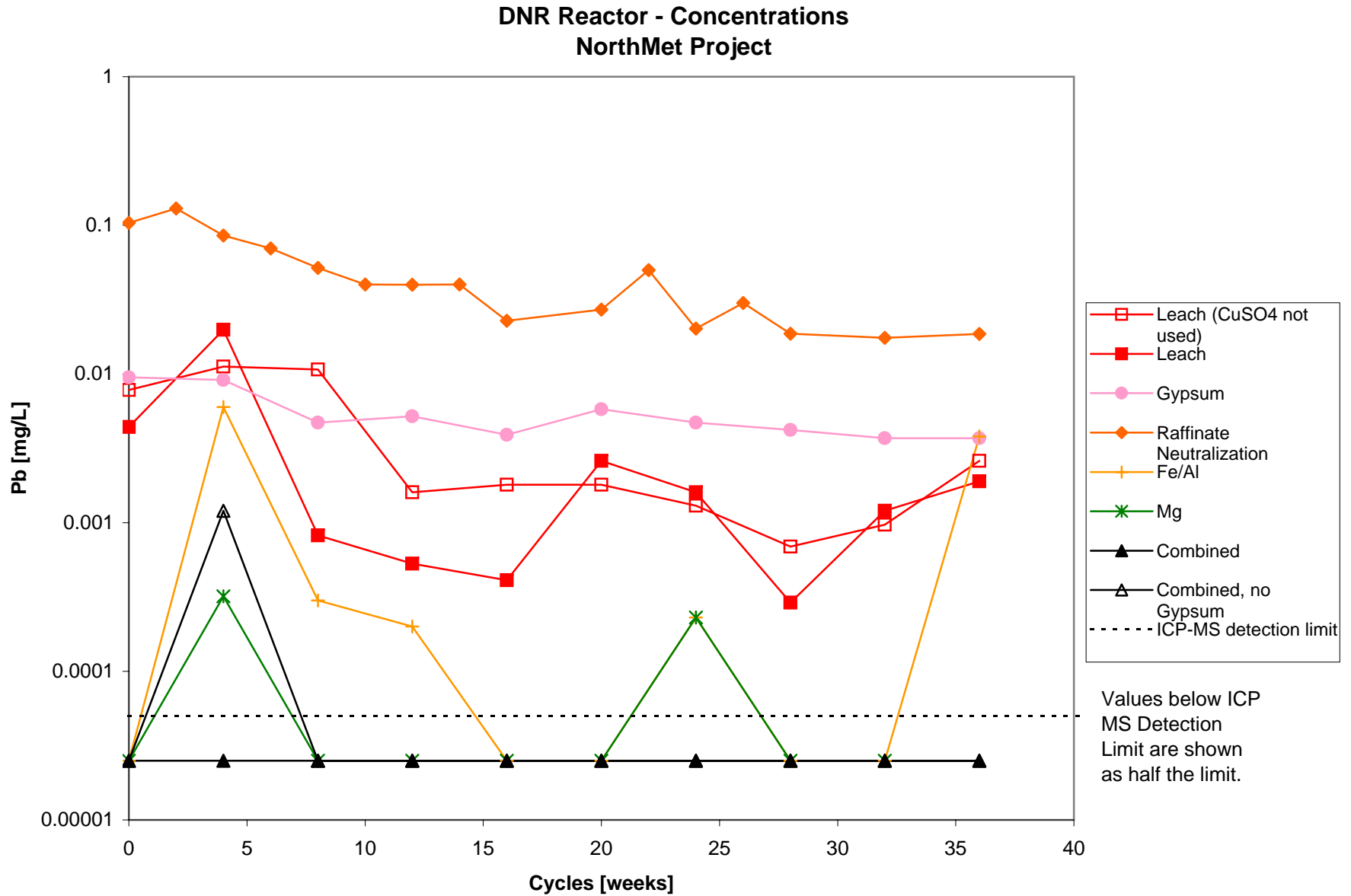
DNR Reactor - Concentrations
NorthMet Project

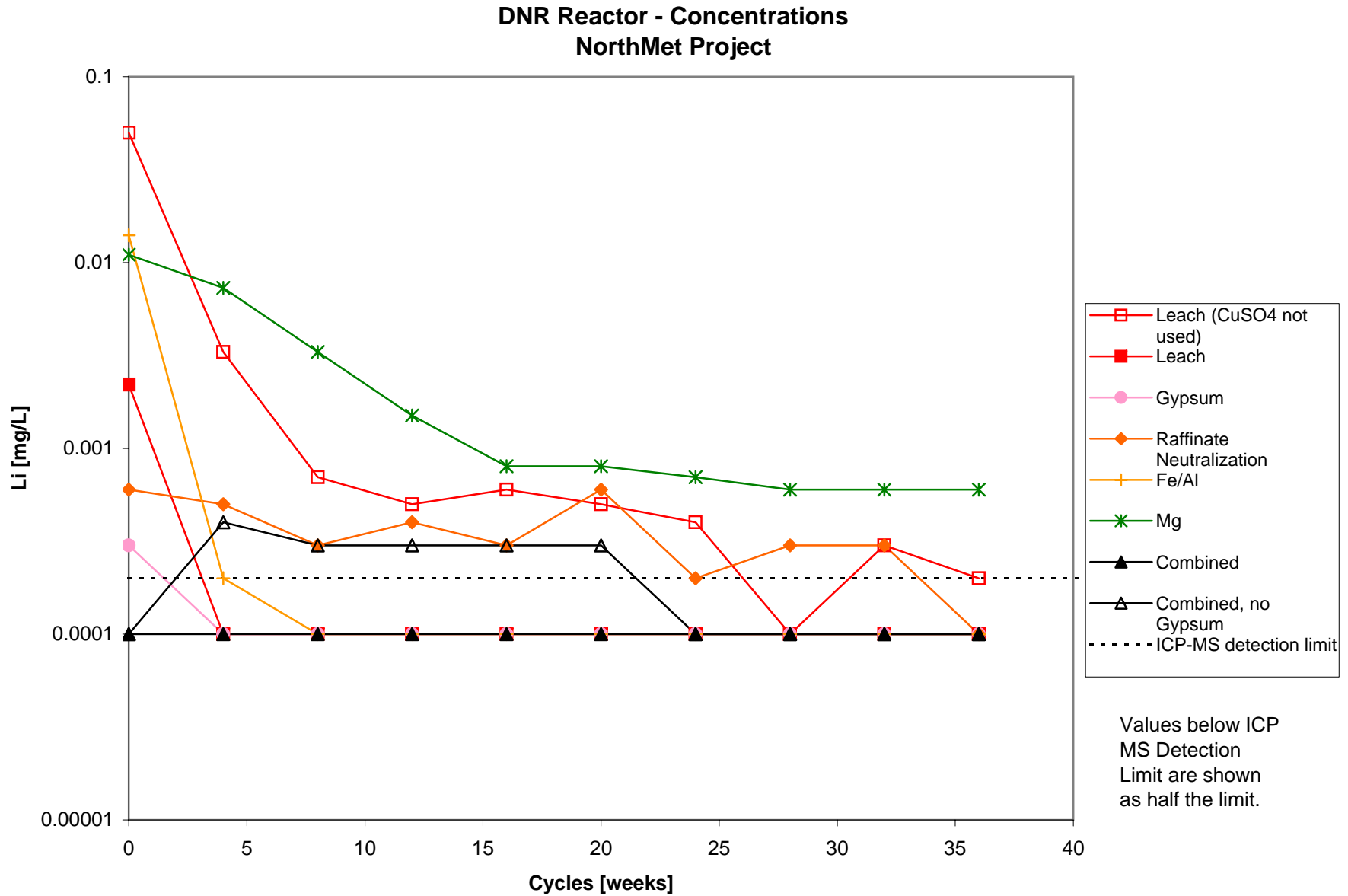


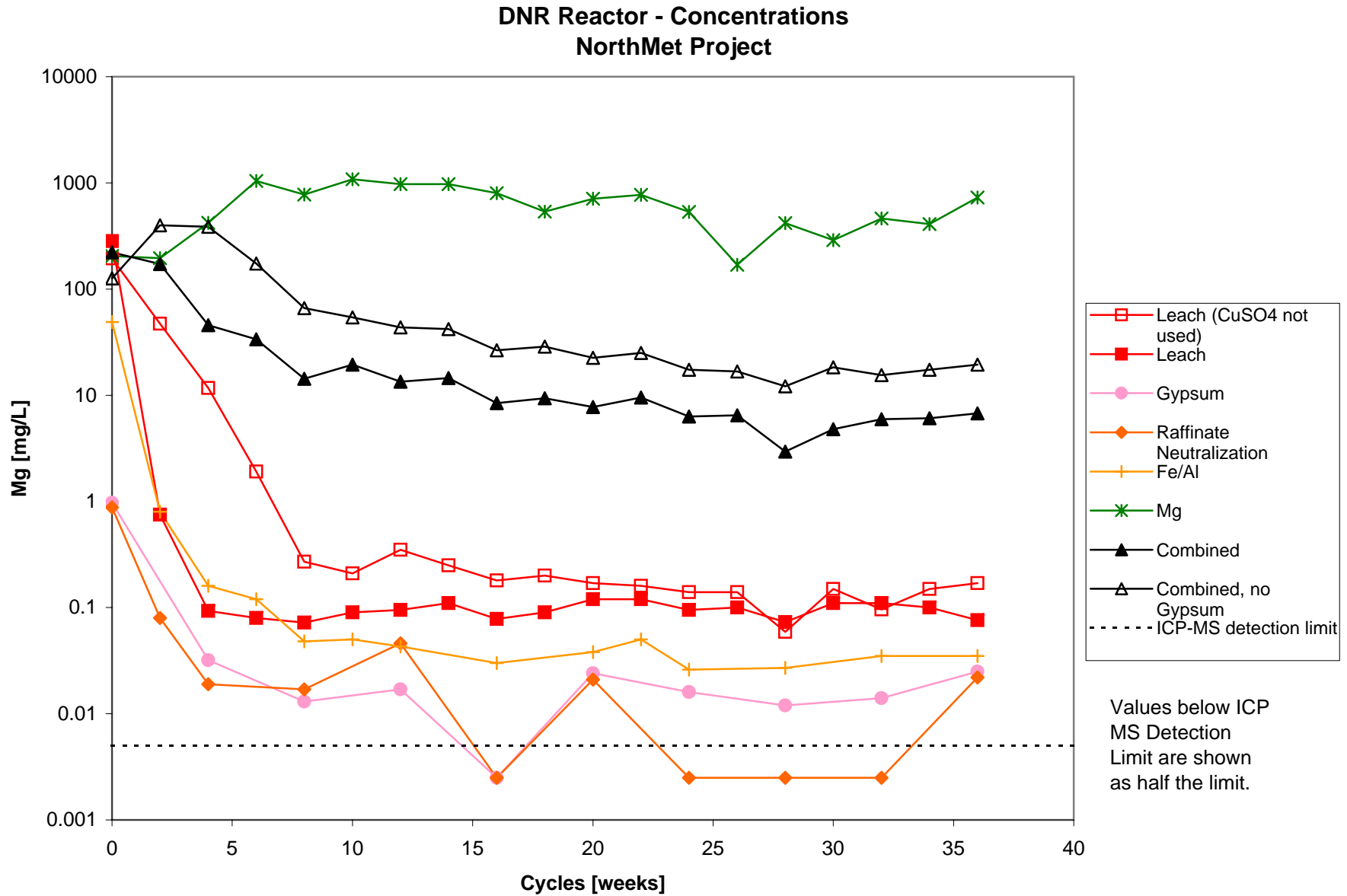


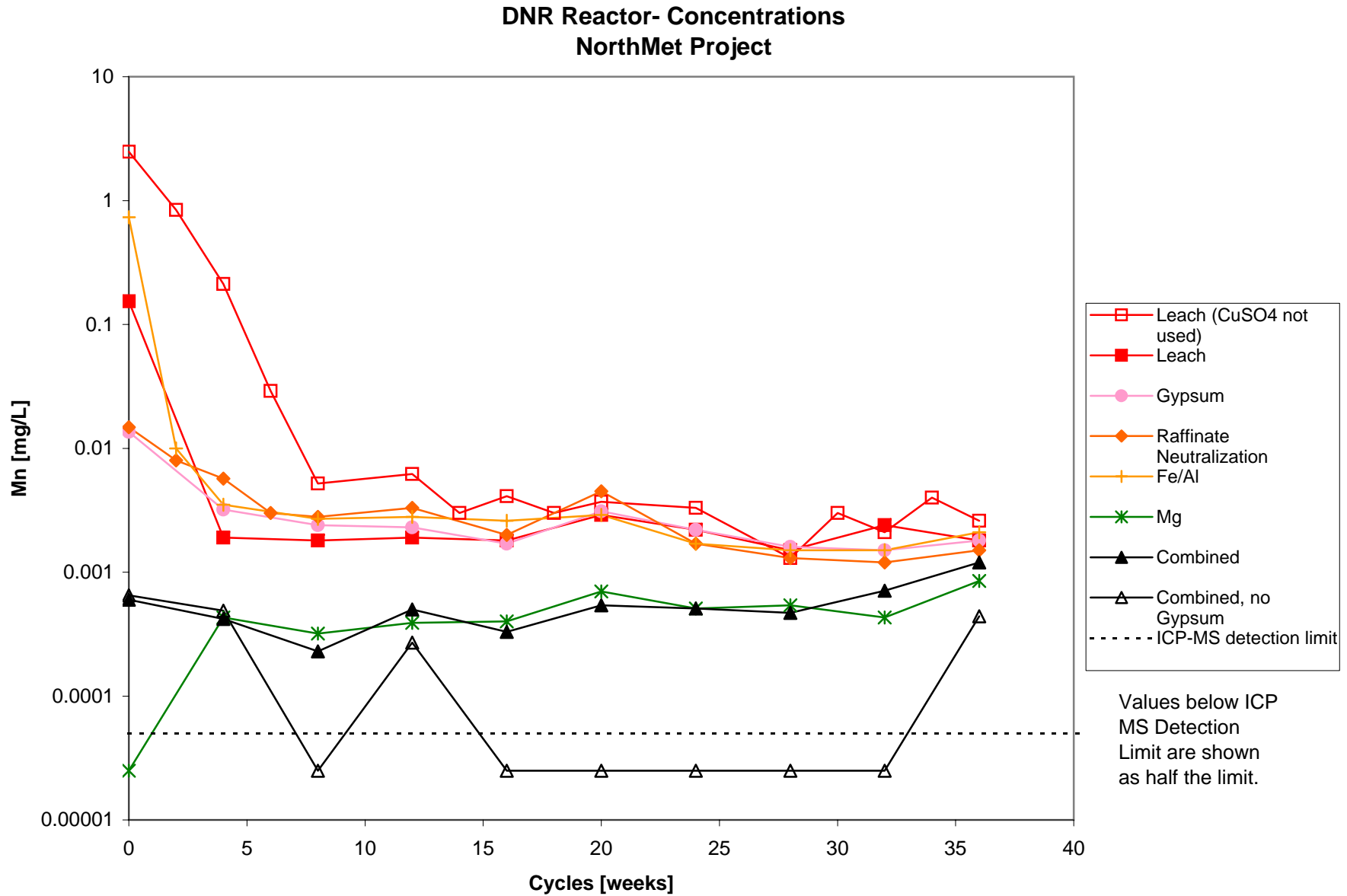
DNR Reactor - Concentrations
NorthMet Project



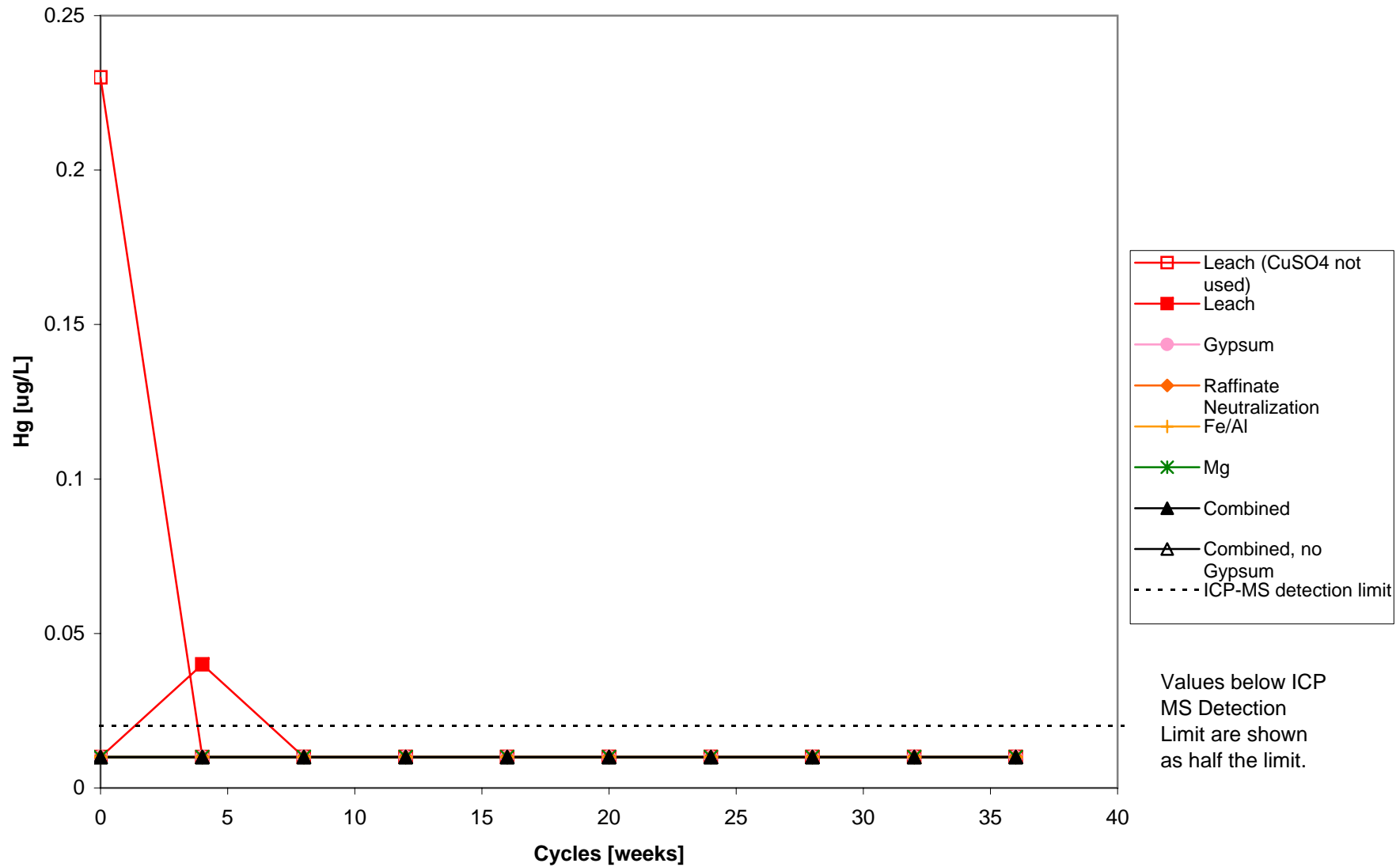


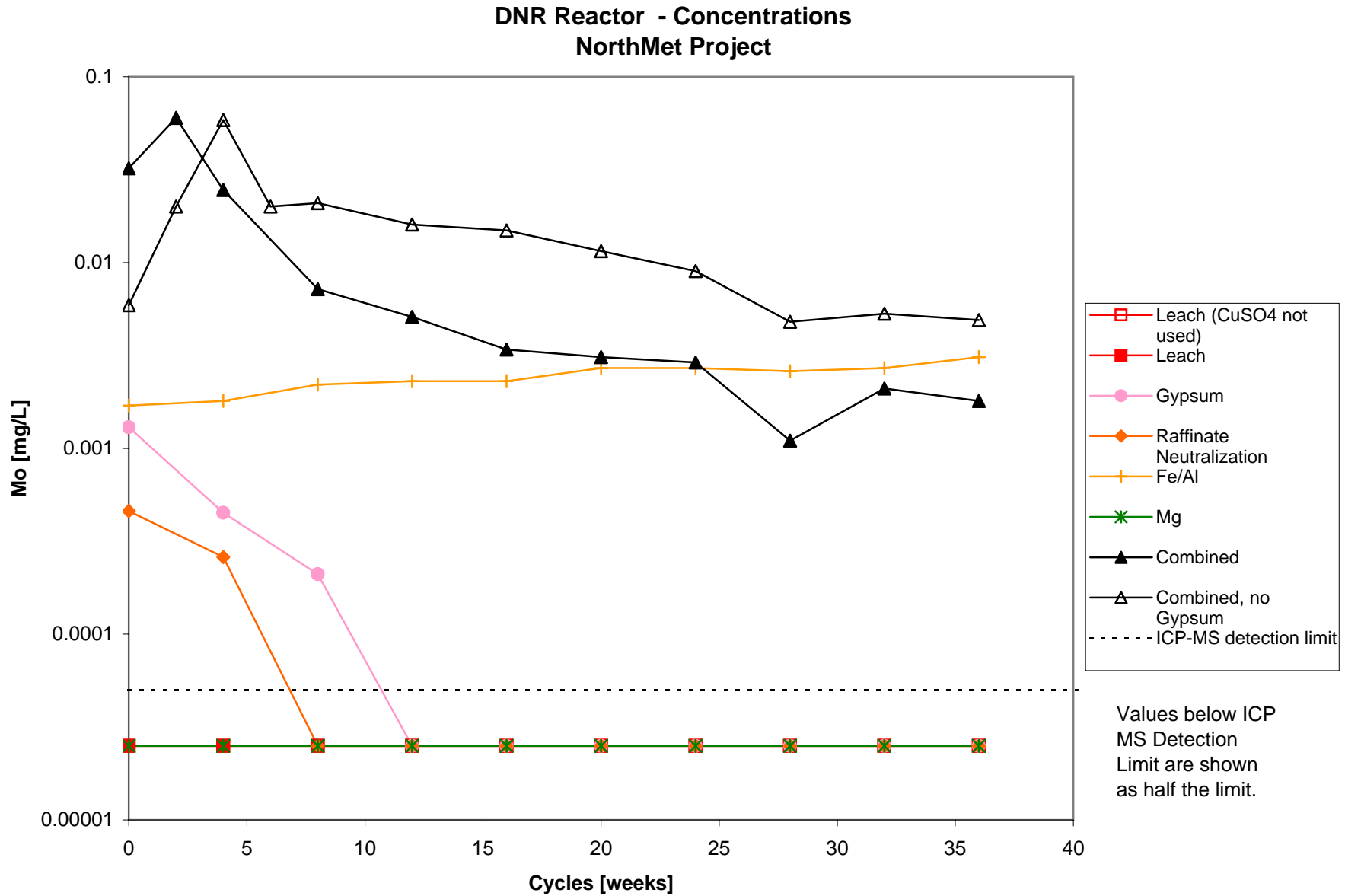


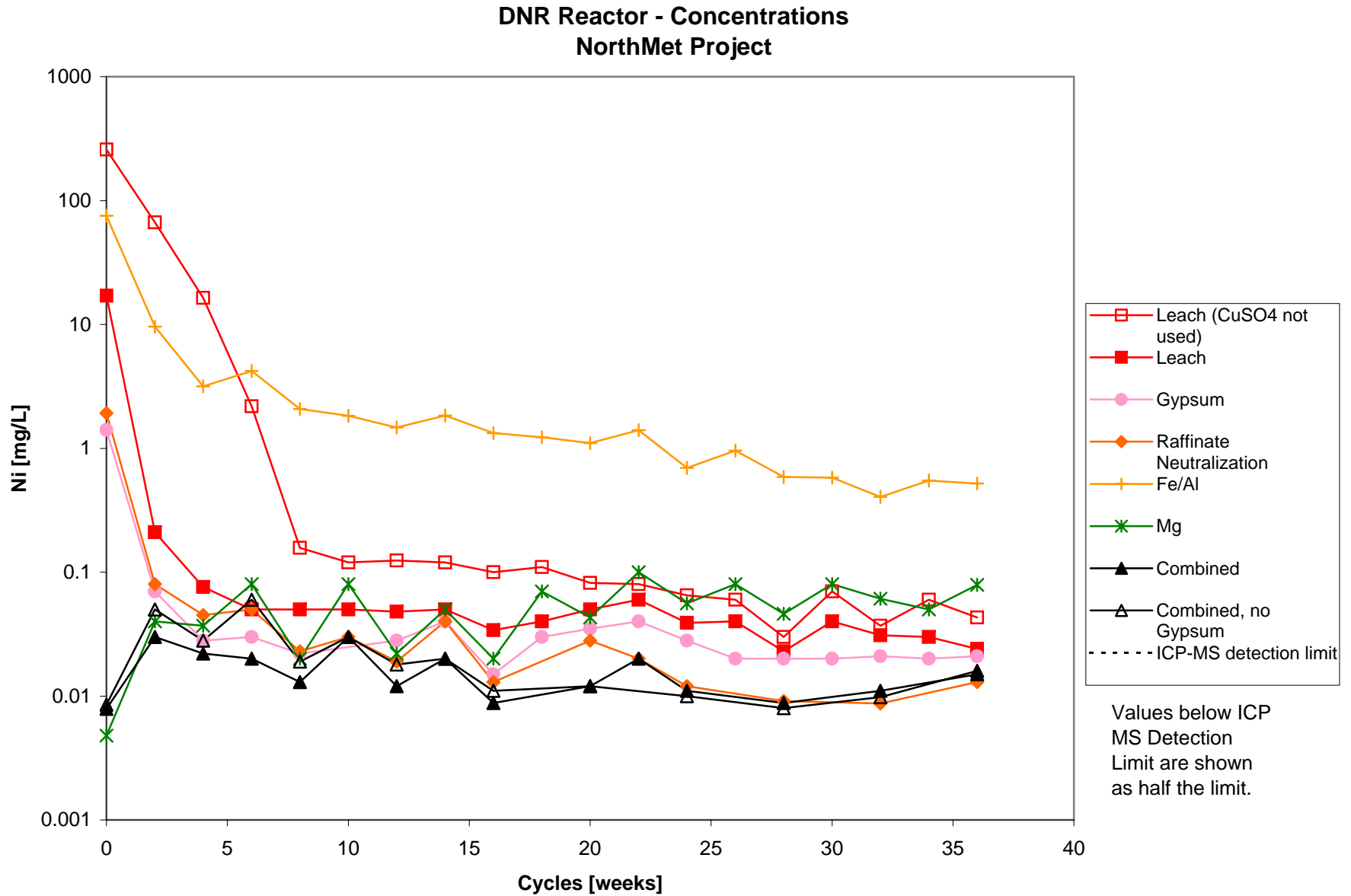




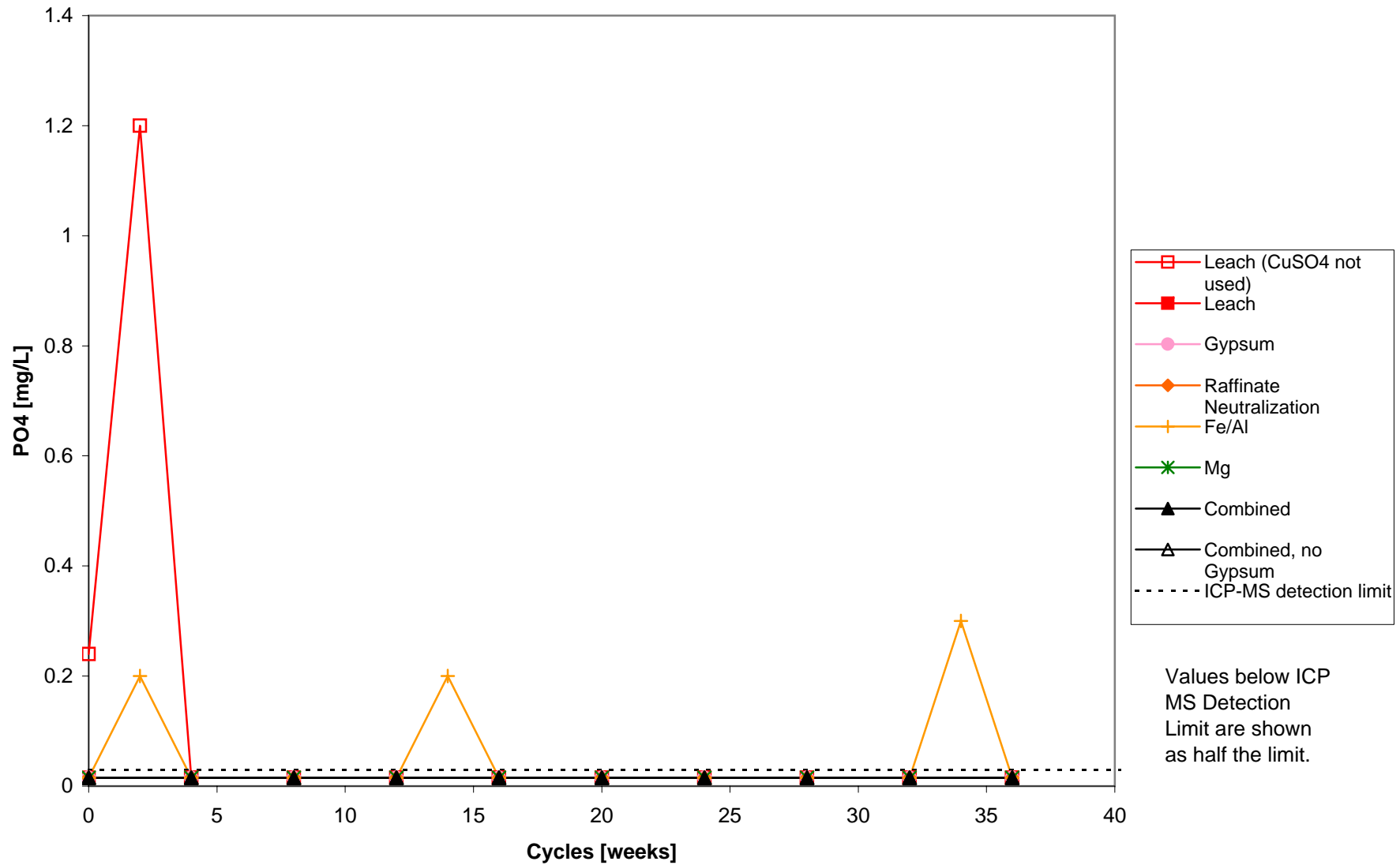
DNR Reactor - Concentrations
NorthMet Project





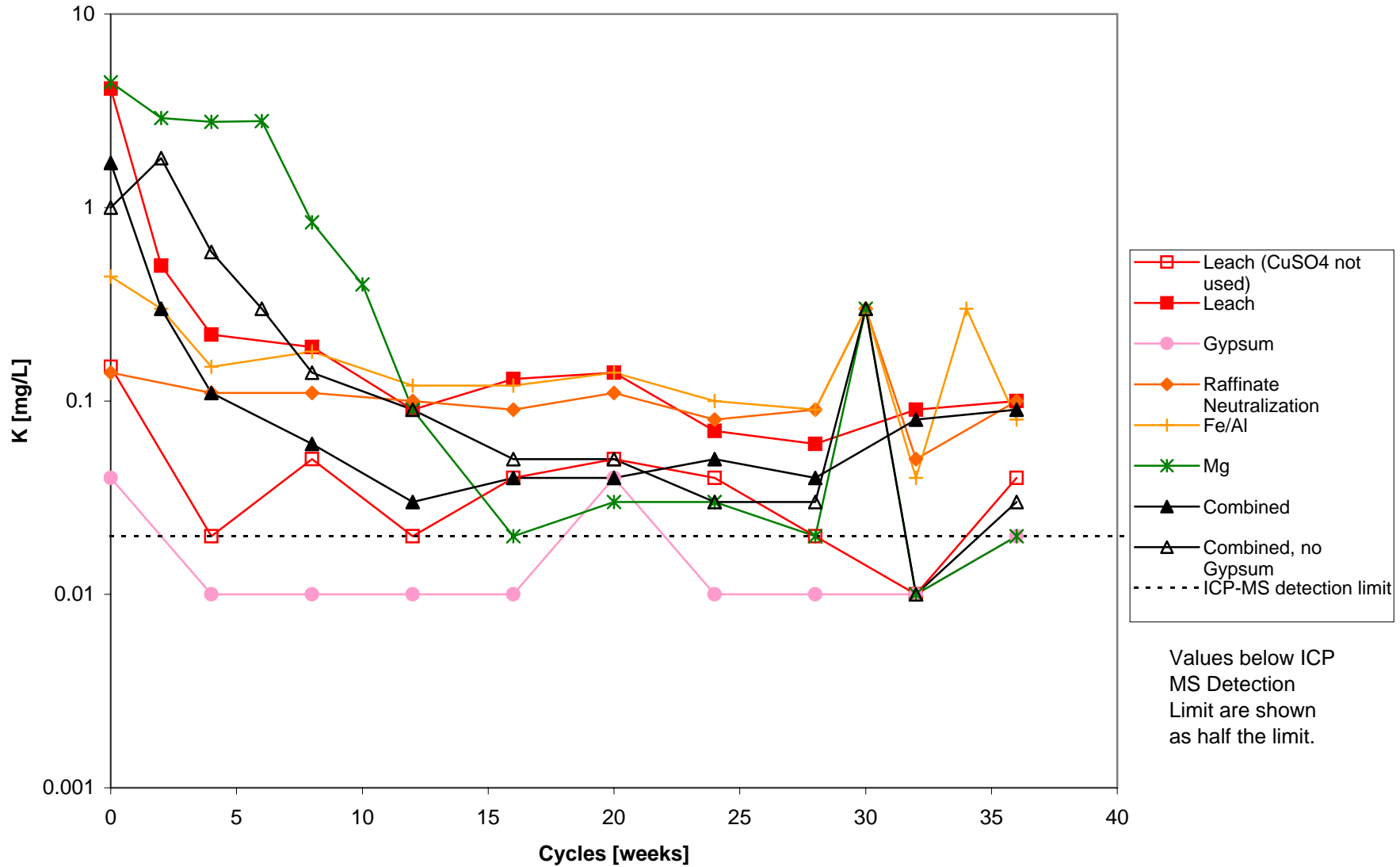


DNR Reactor - Concentrations
NorthMet Project



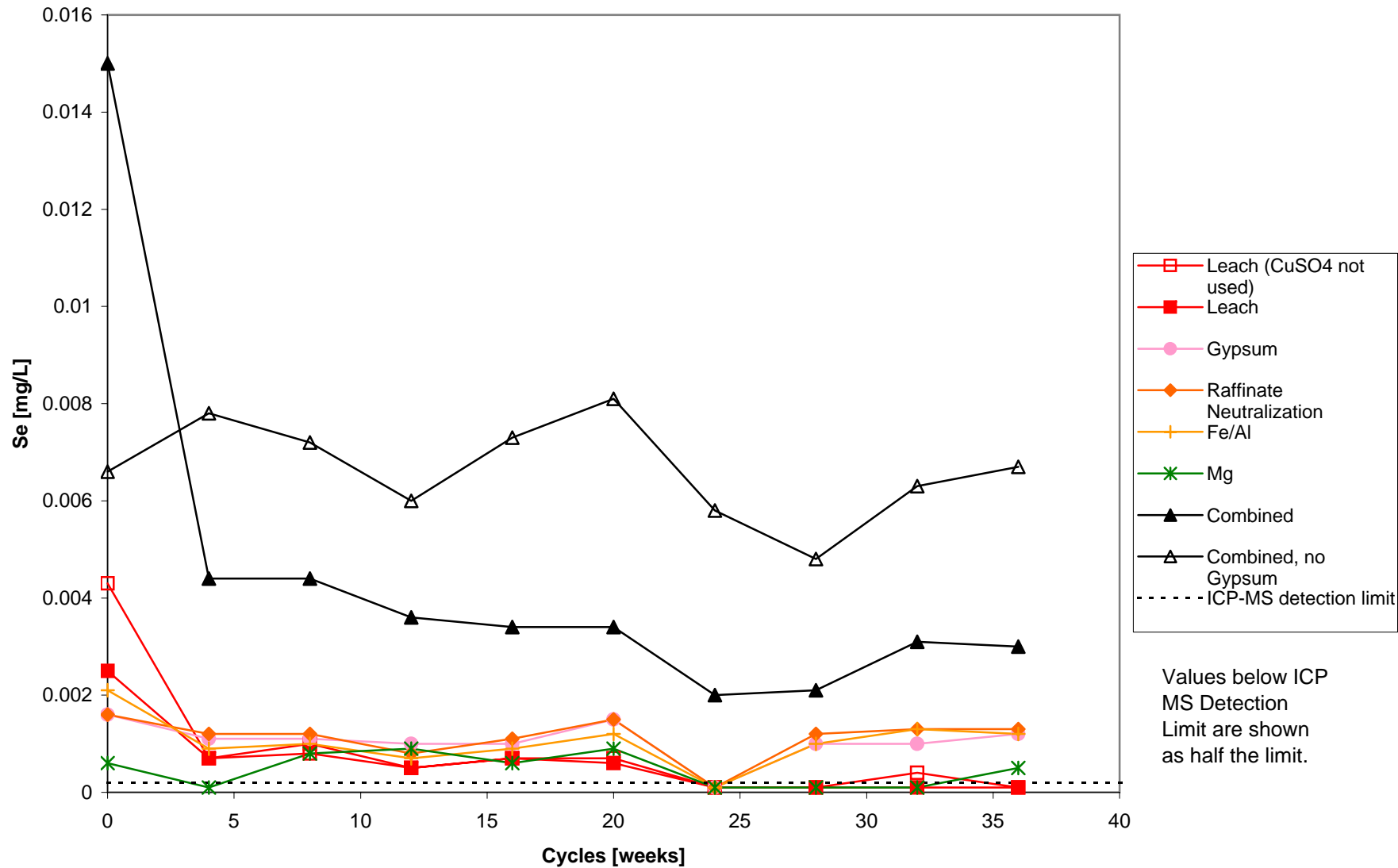
Values below ICP
MS Detection
Limit are shown
as half the limit.

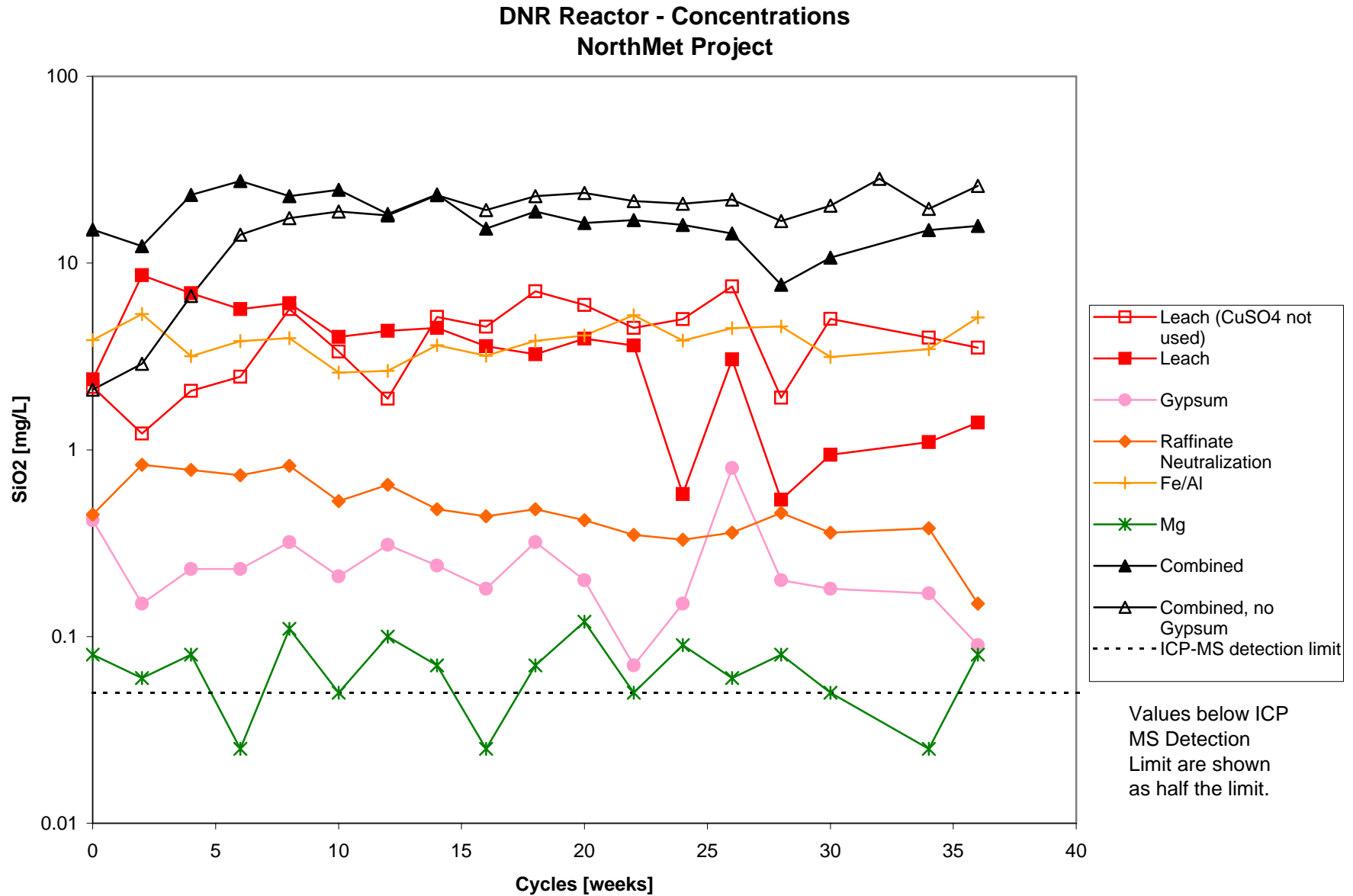
DNR Reactor - Concentrations
NorthMet Project



Values below ICP
MS Detection
Limit are shown
as half the limit.

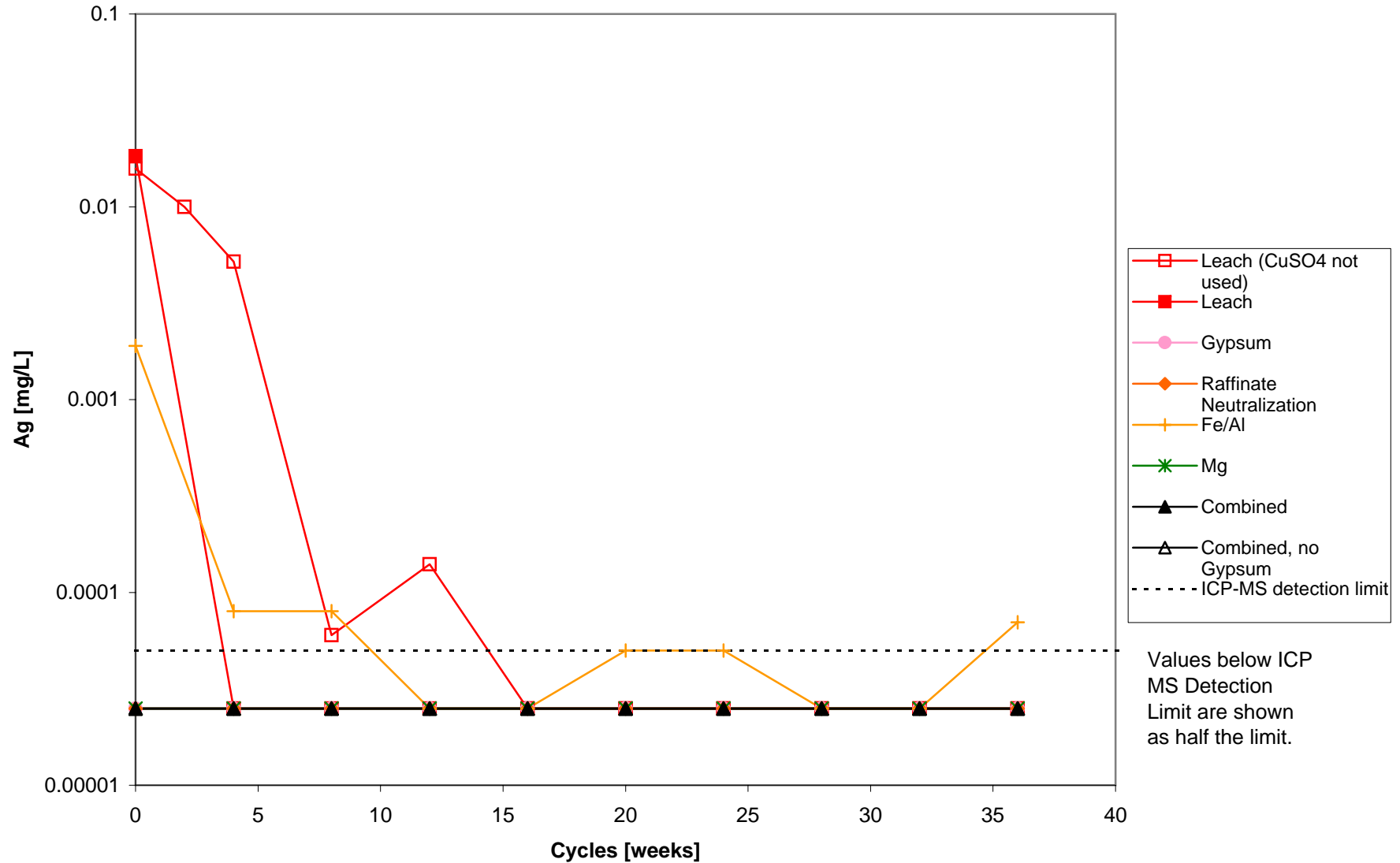
DNR Reactor - Concentrations
NorthMet Project



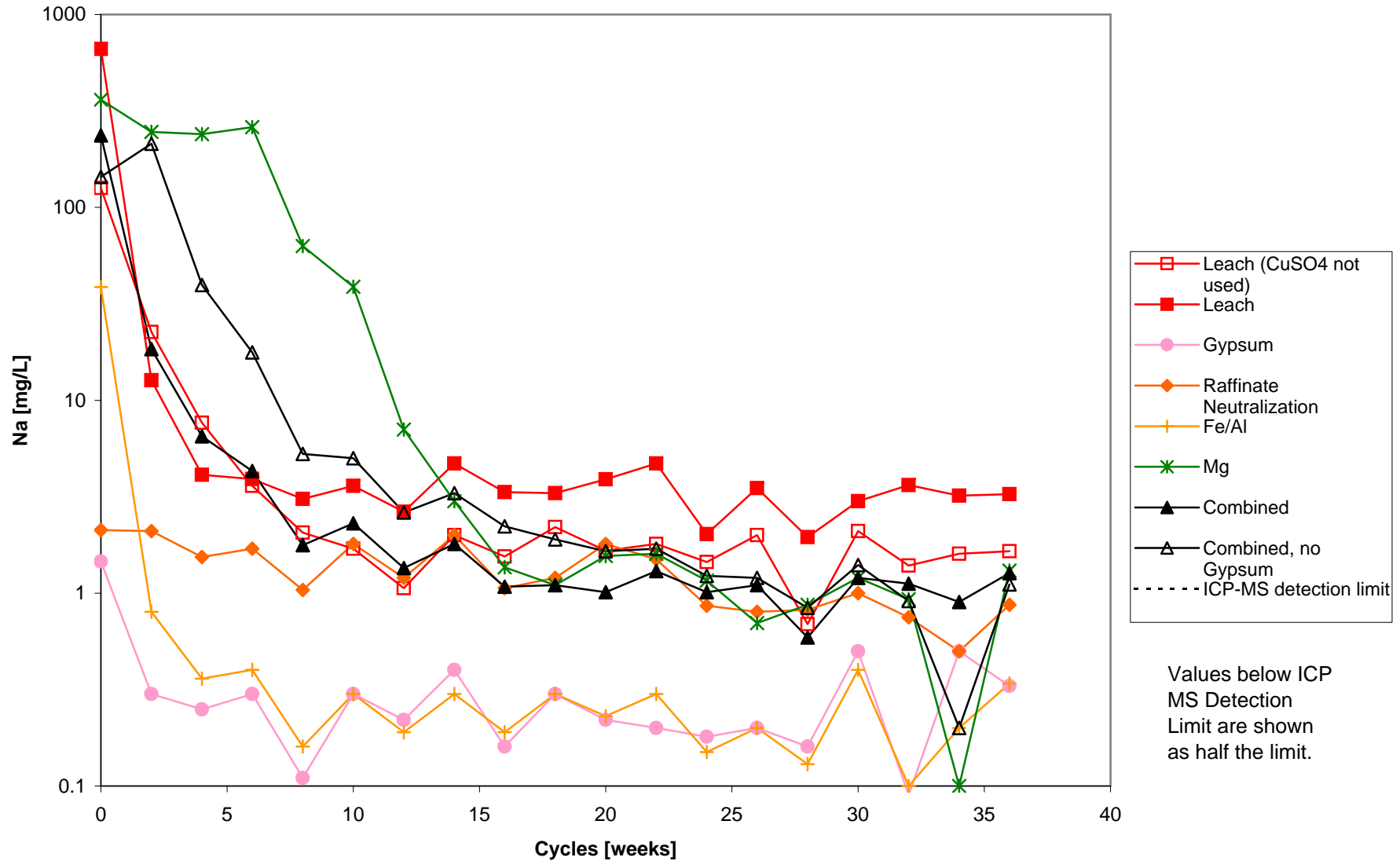


Values below ICP
MS Detection
Limit are shown
as half the limit.

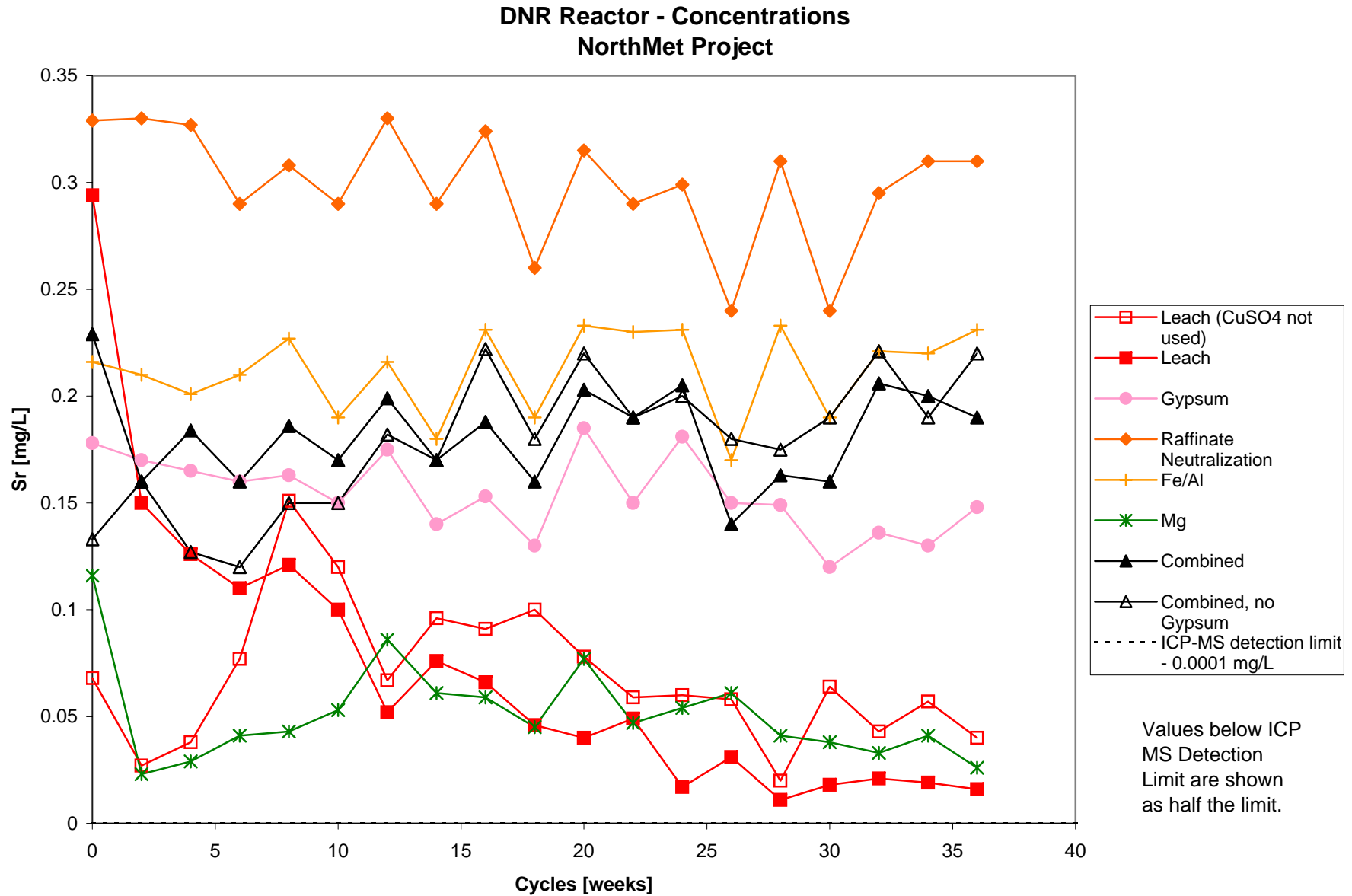
DNR Reactor - Concentrations
NorthMet Project



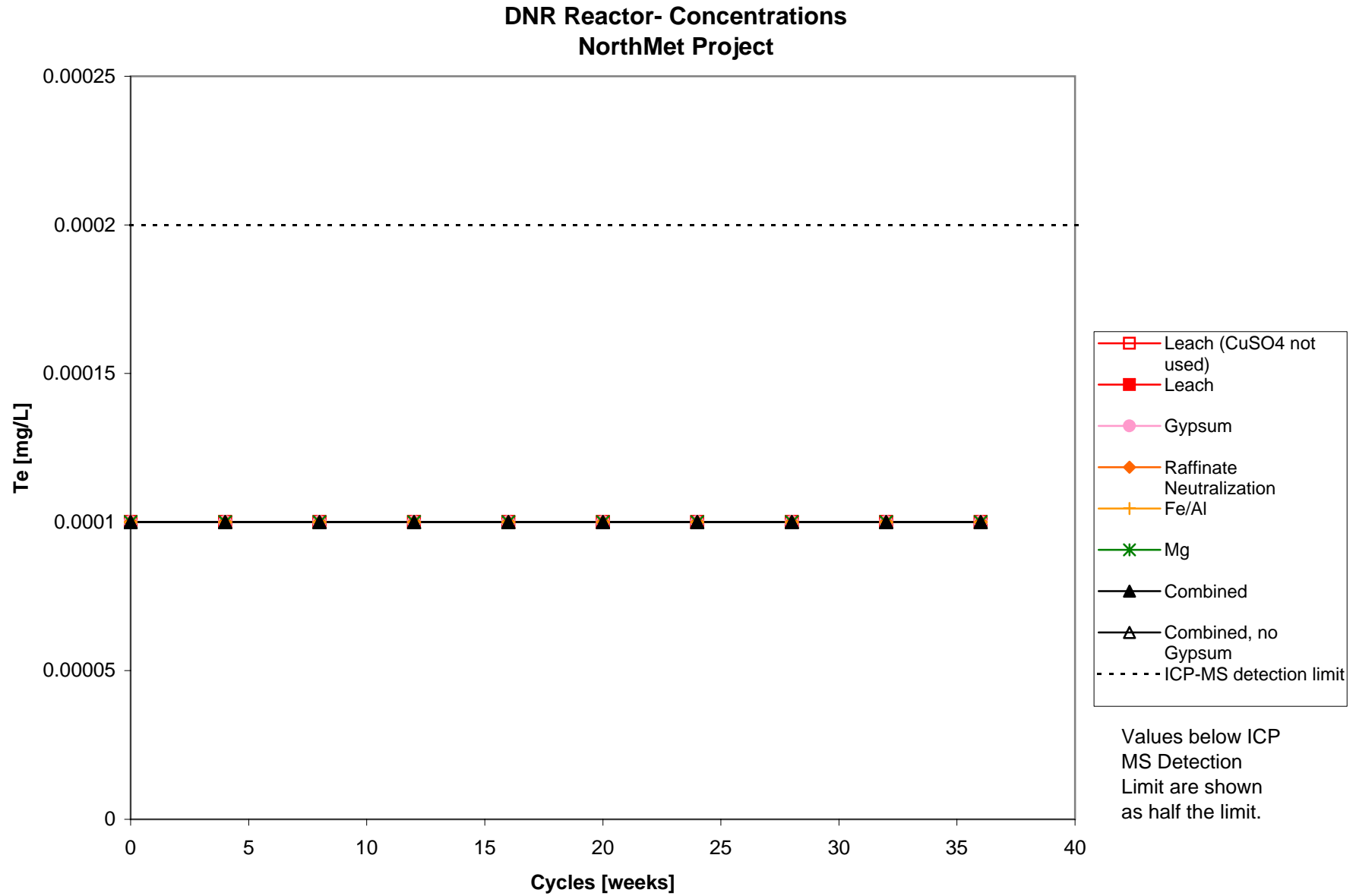
DNR Reactor - Concentrations
NorthMet Project

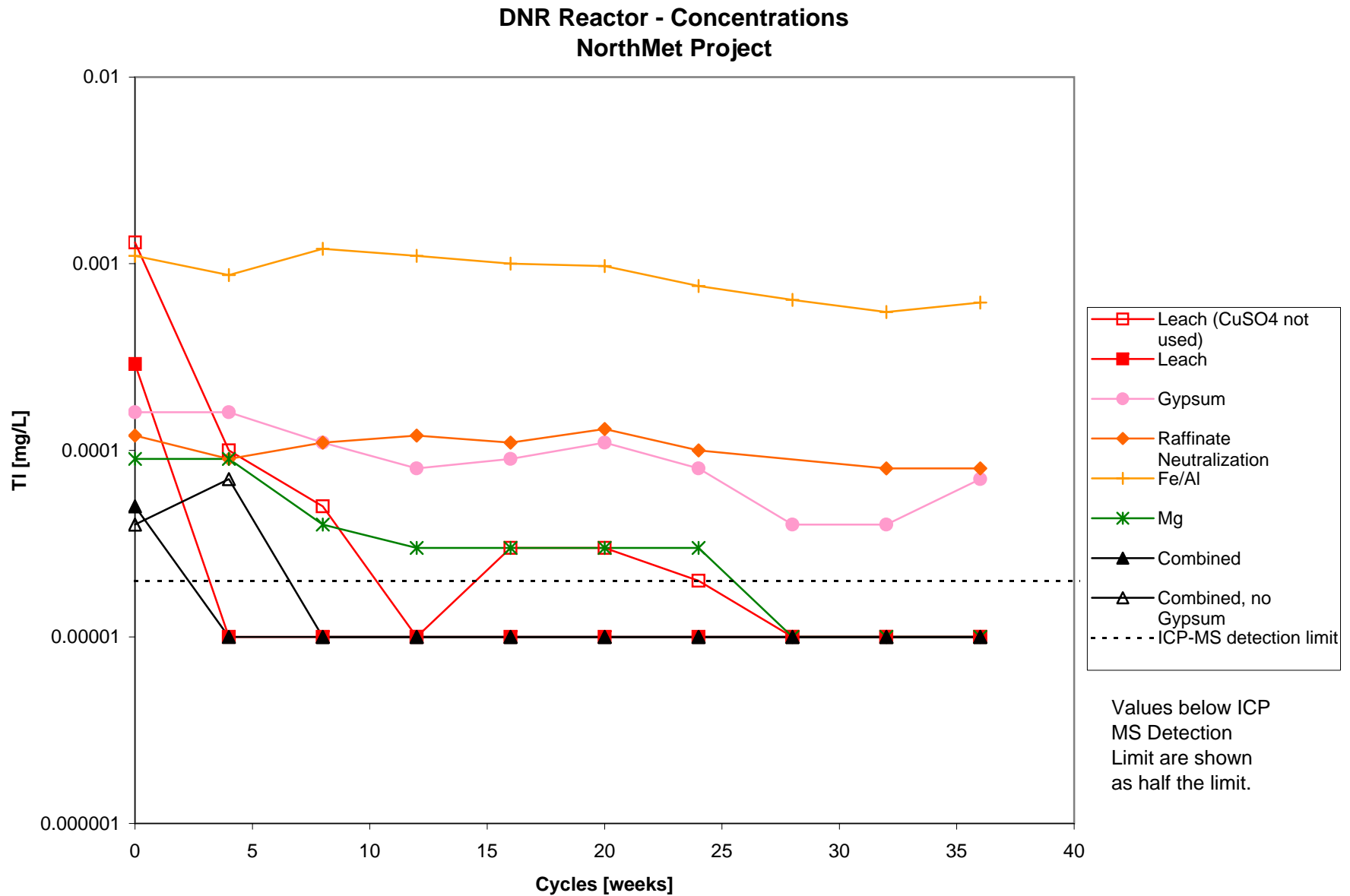


Values below ICP
MS Detection
Limit are shown
as half the limit.

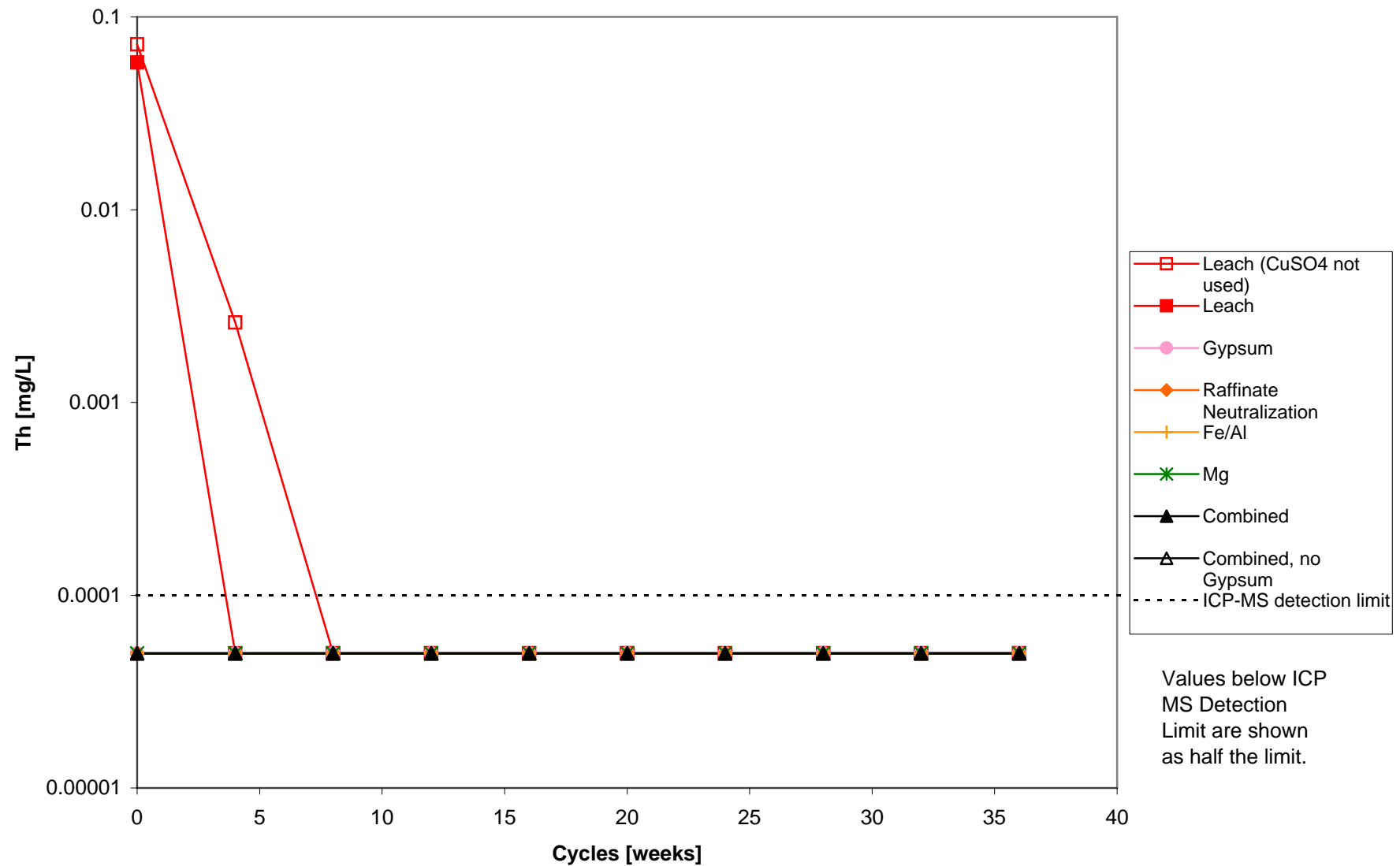


Values below ICP MS Detection Limit are shown as half the limit.



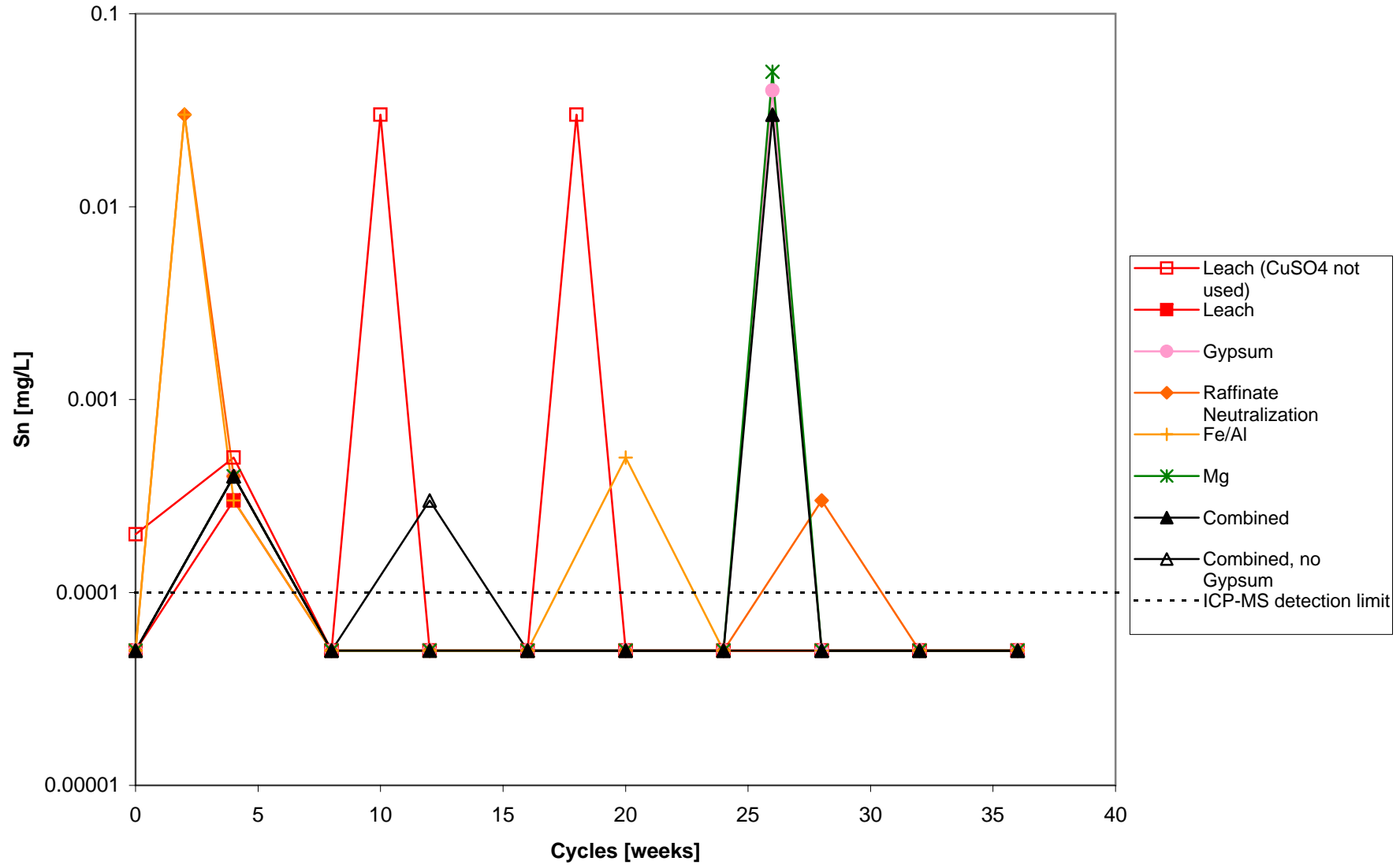


DNR Reactor - Concentrations
NorthMet Project

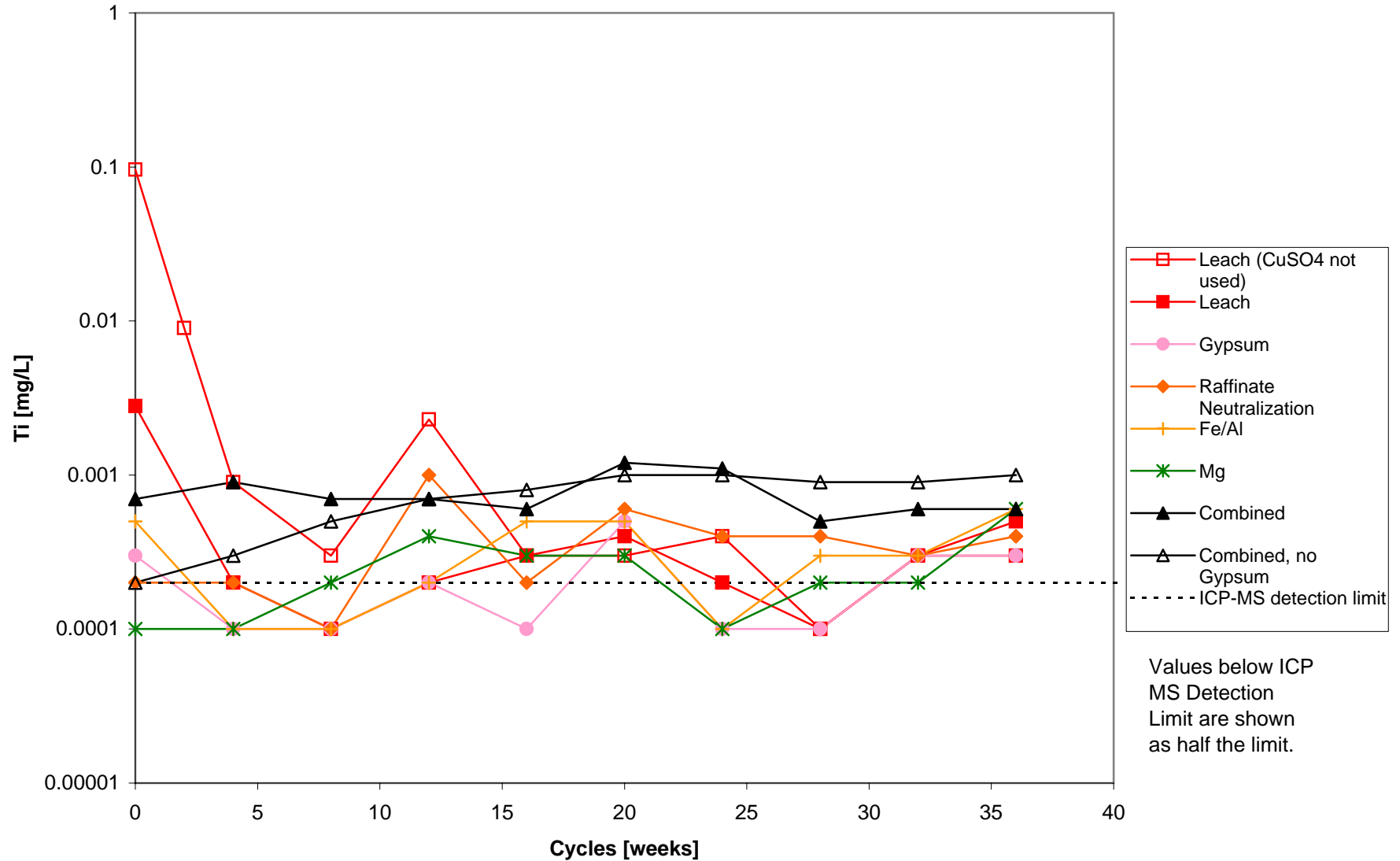


Values below ICP-MS Detection Limit are shown as half the limit.

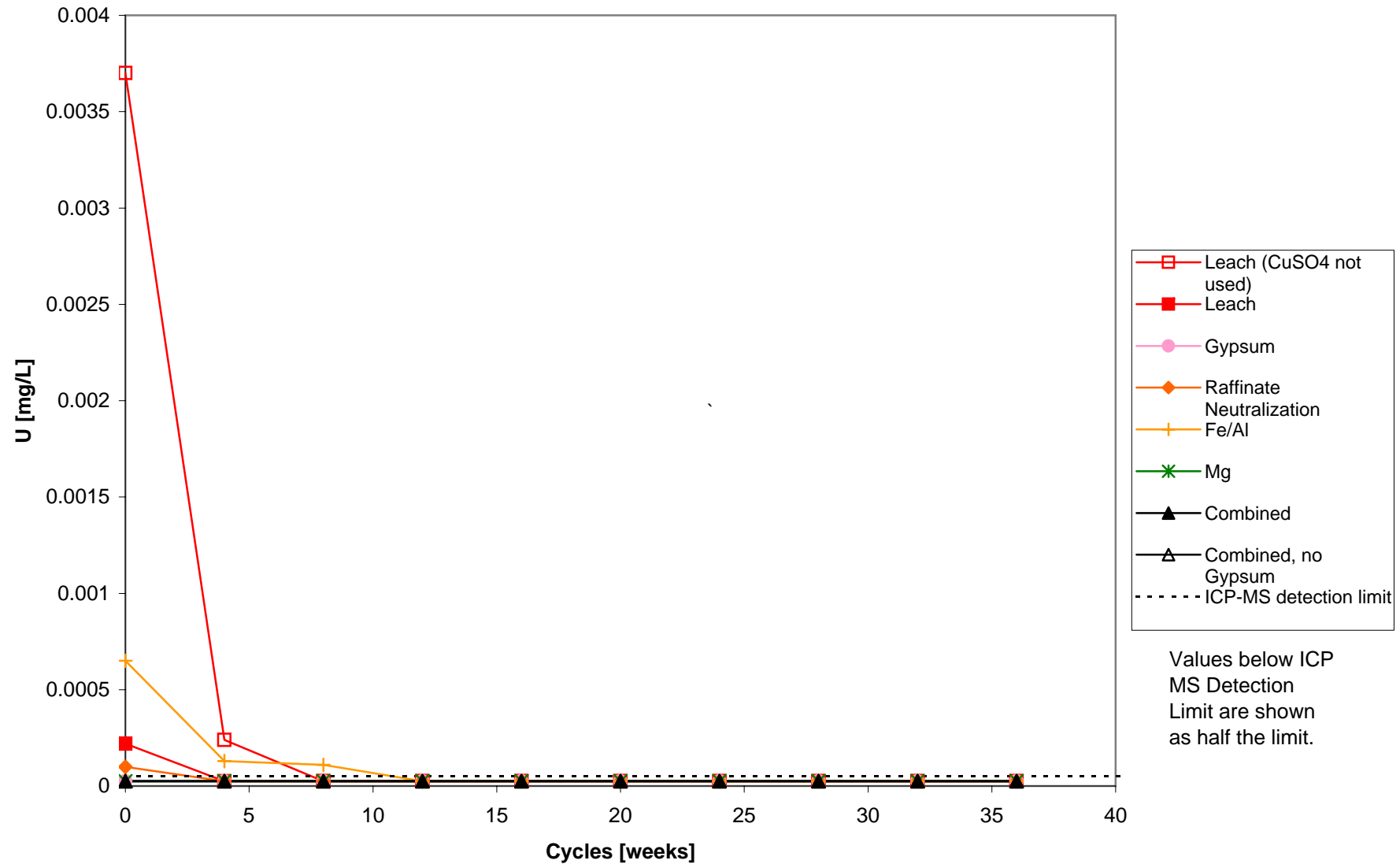
DNR Reactor - Concentrations
NorthMet Project

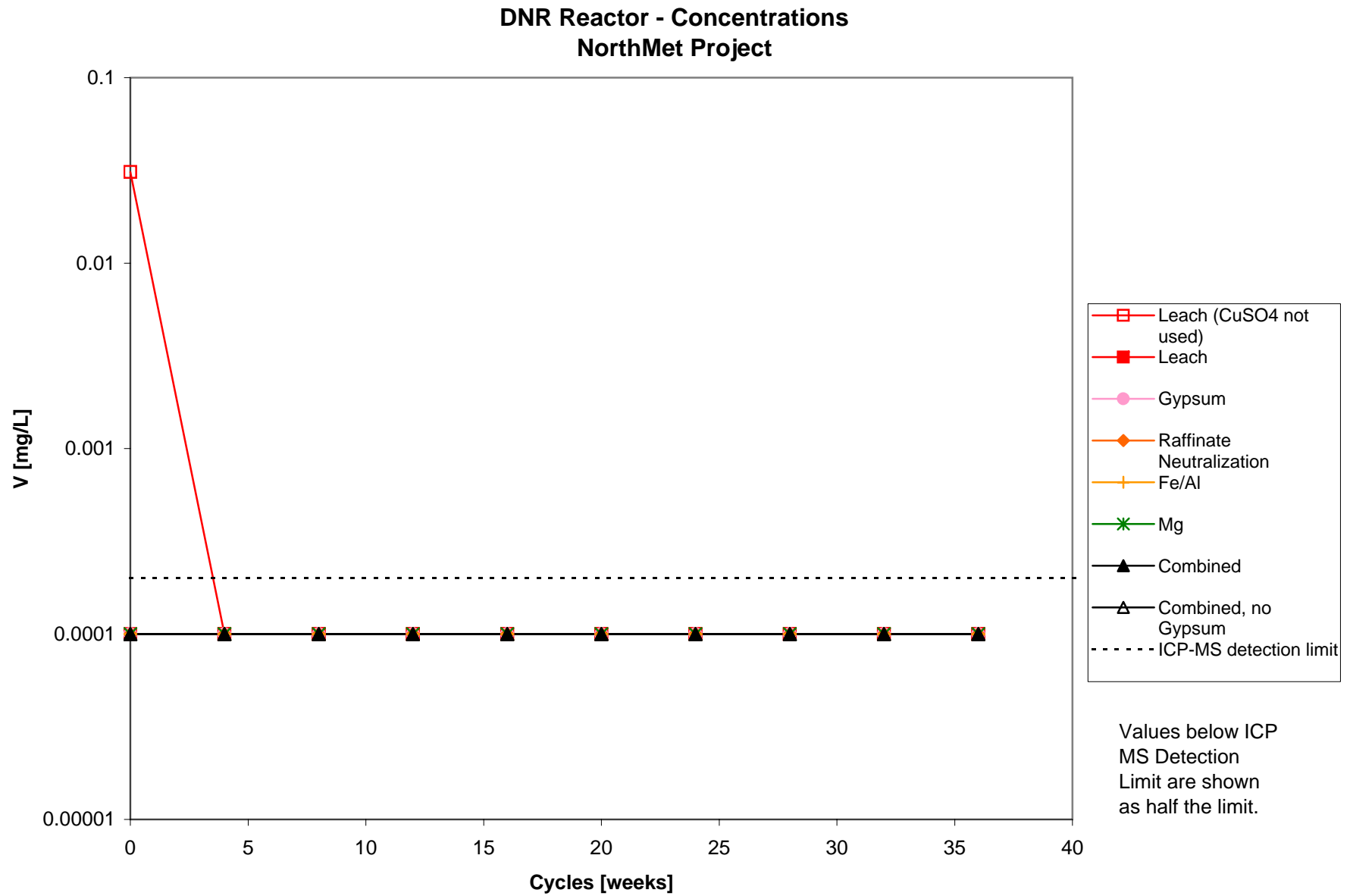


DNR Reactor - Concentrations
NorthMet Project

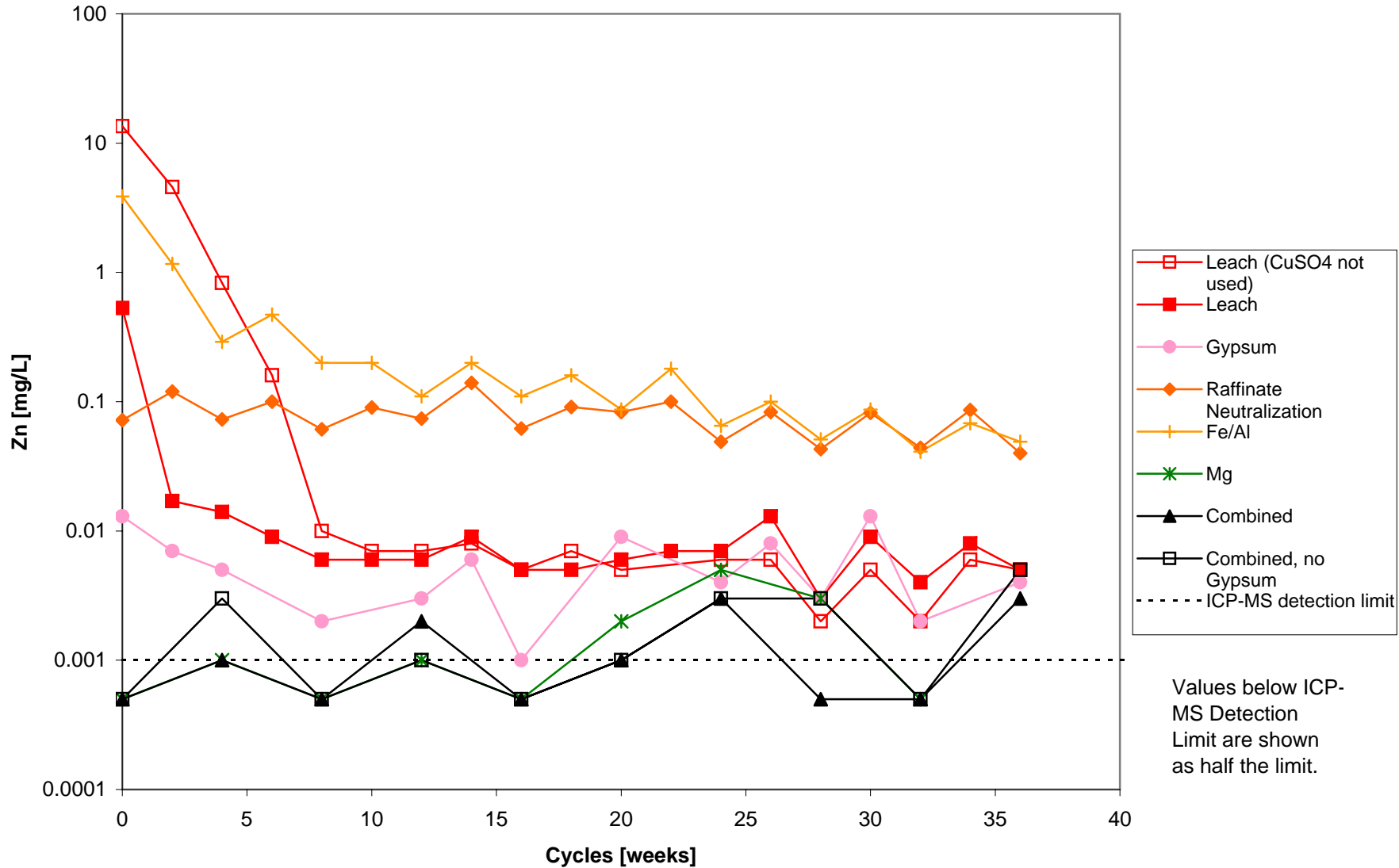


DNR Reactor - Concentrations
NorthMet Project

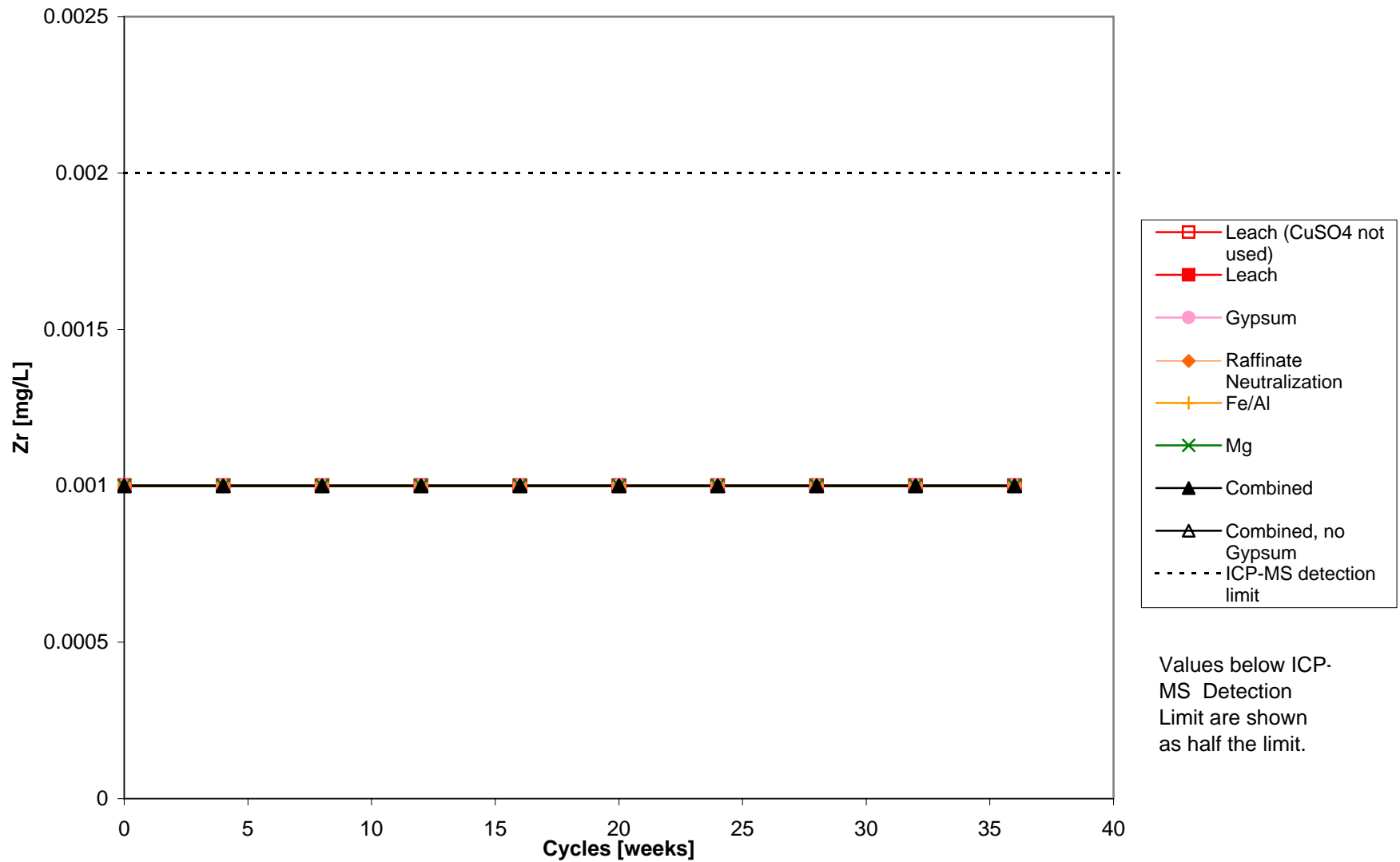




DNR Reactor- Concentrations
NorthMet Project



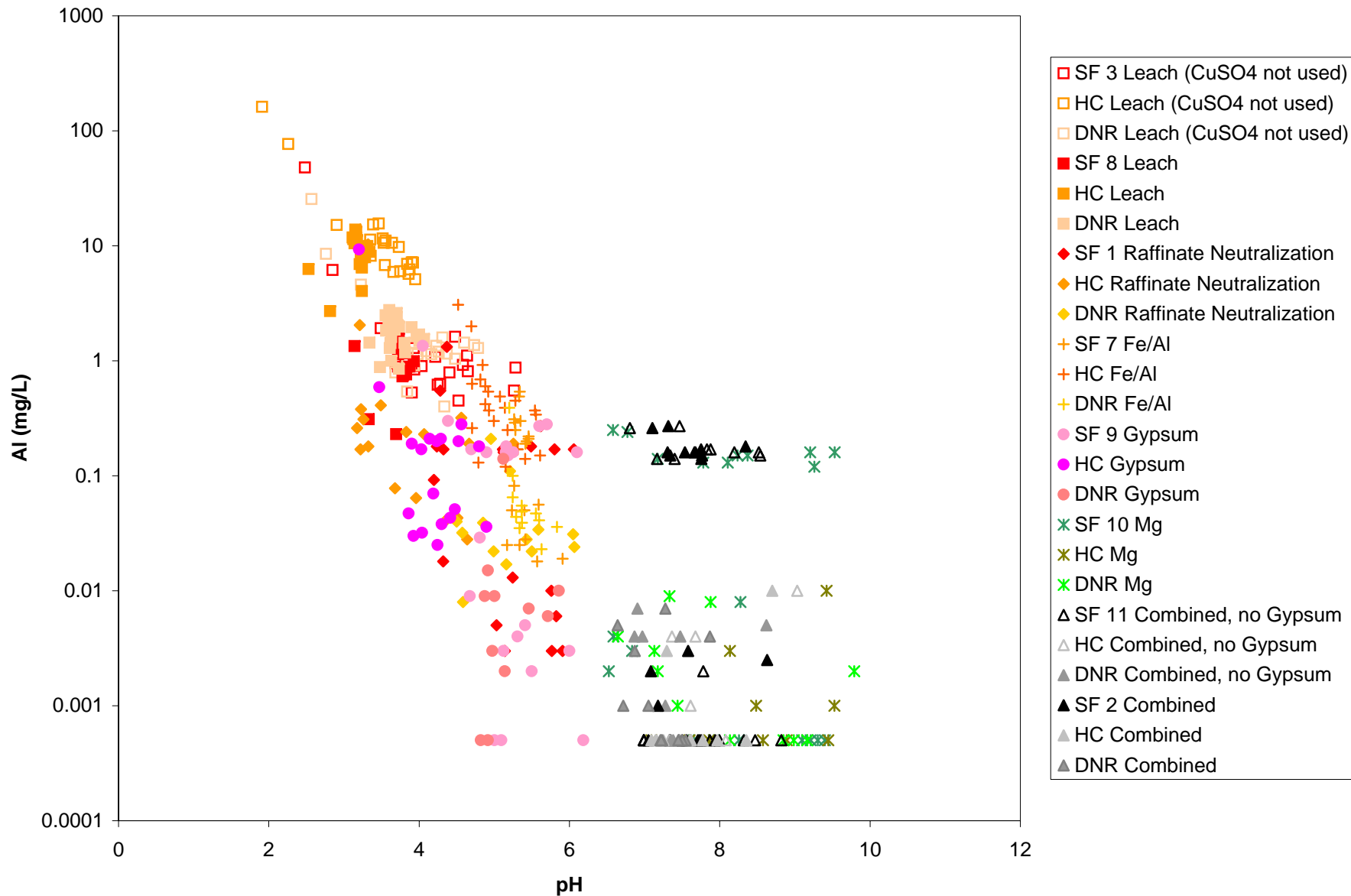
DNR Reactor - Concentrations
NorthMet Project



Appendix D
Scatter Plots Showing Metal Concentrations Compared to pH

Appendix D
Scatter Plots Showing Metal Concentrations Compared to pH

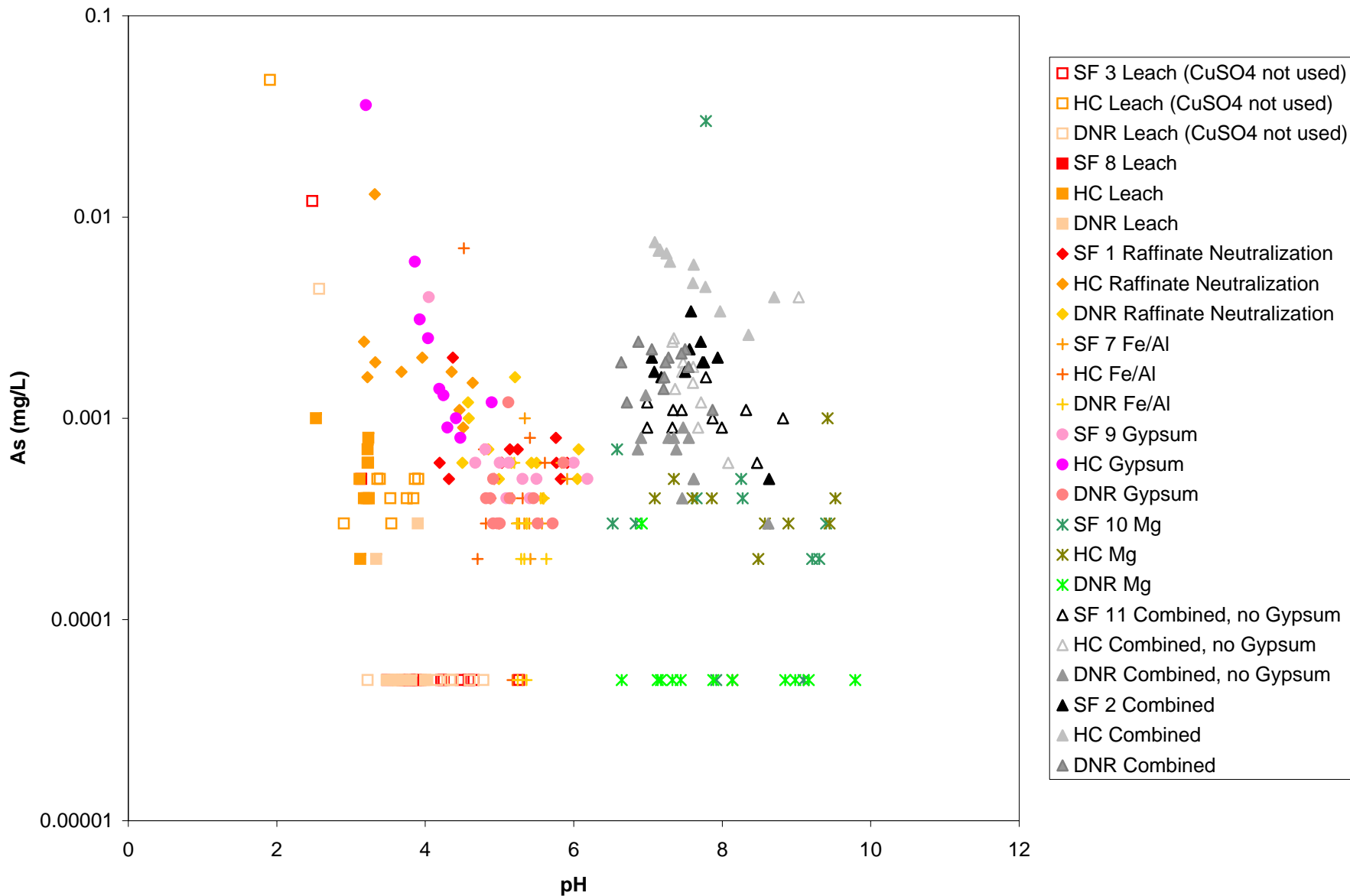
Graph D.1



M(pH)

Appendix D
Scatter Plots Showing Metal Concentrations Compared to pH

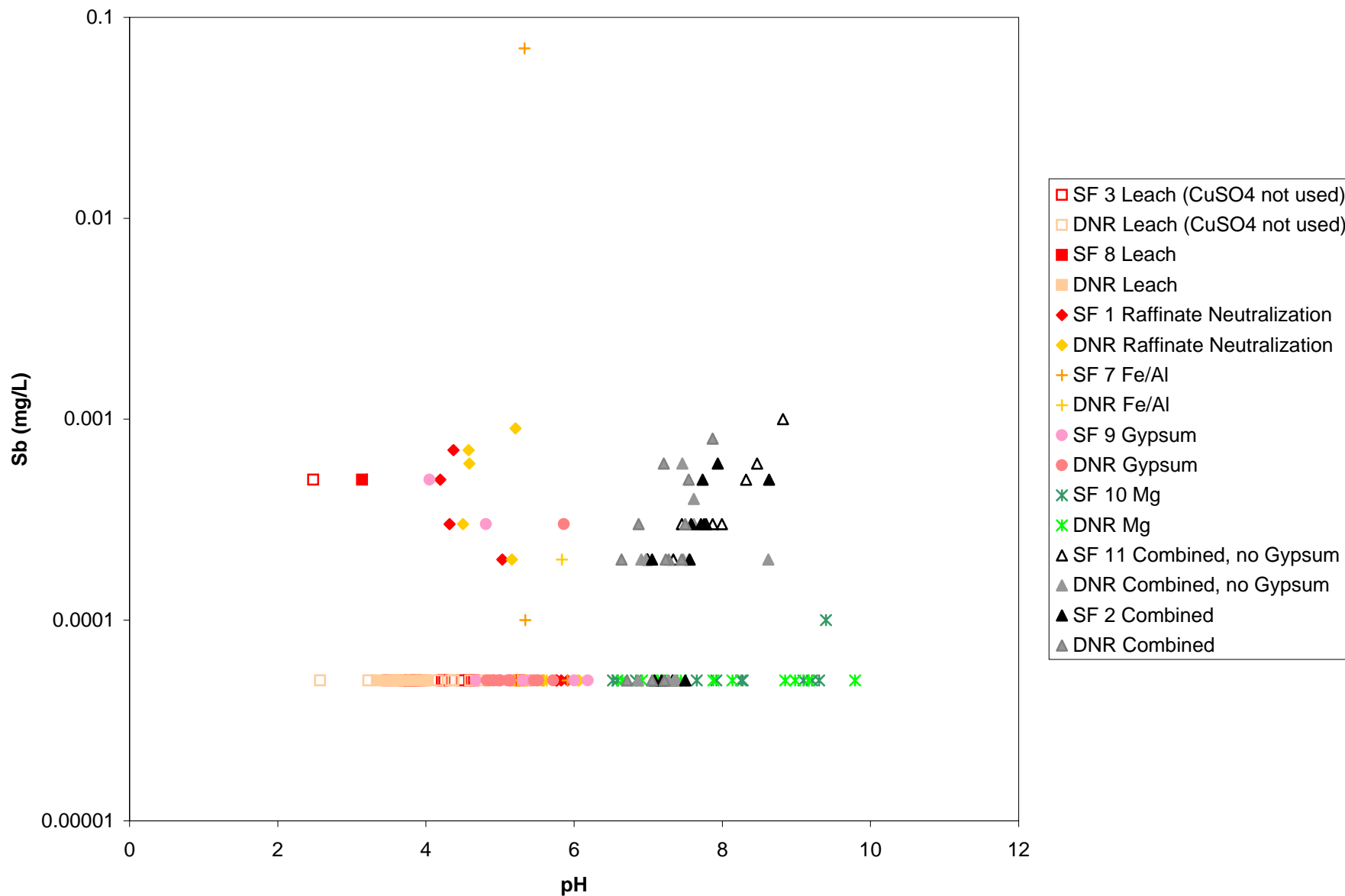
Graph D.2



M(pH)

Appendix D
Scatter Plots Showing Metal Concentrations Compared to pH

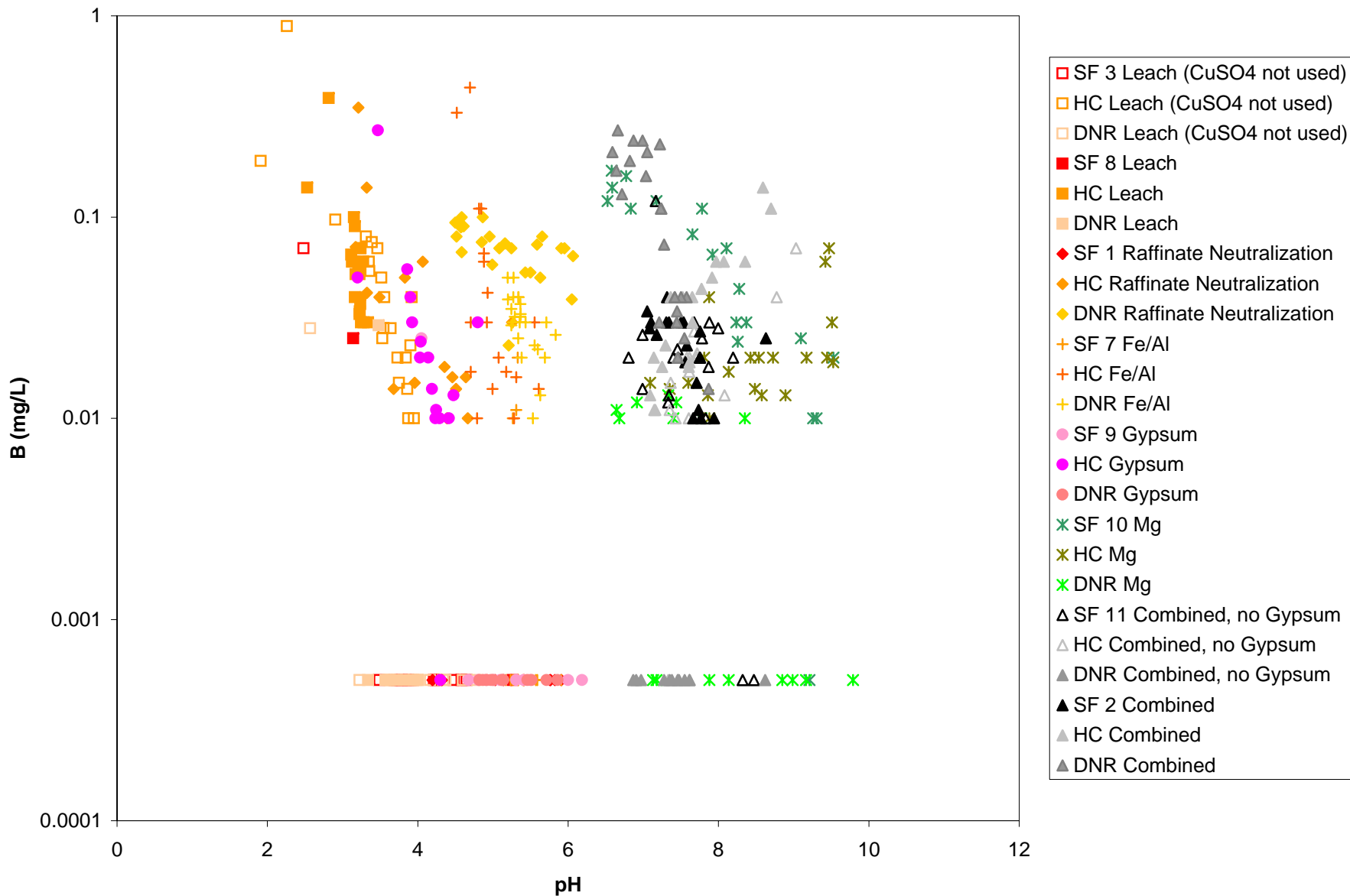
Graph D.3



M(pH)

Appendix D
Scatter Plots Showing Metal Concentrations Compared to pH

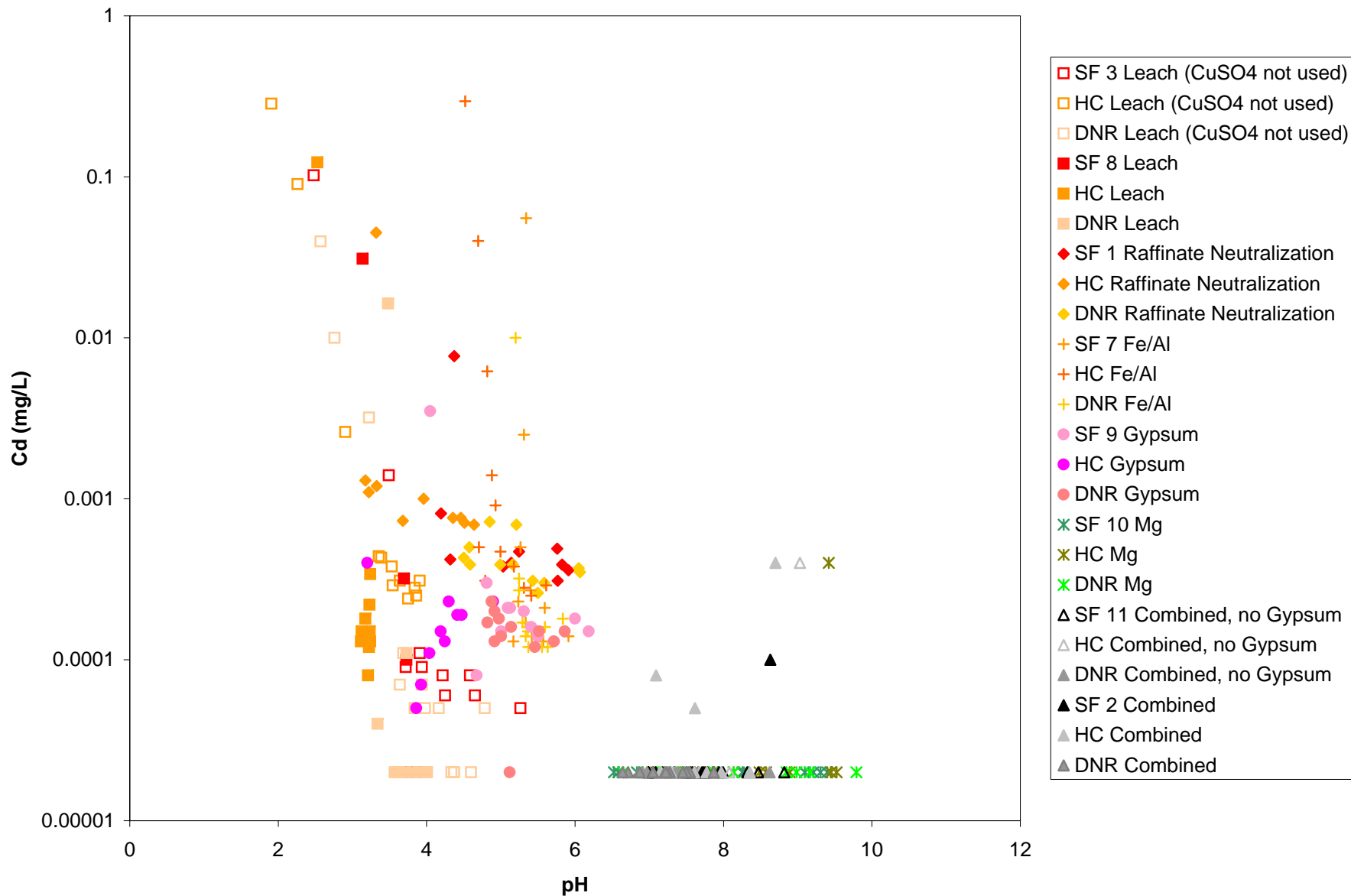
Graph D.4



M(pH)

Appendix D
Scatter Plots Showing Metal Concentrations Compared to pH

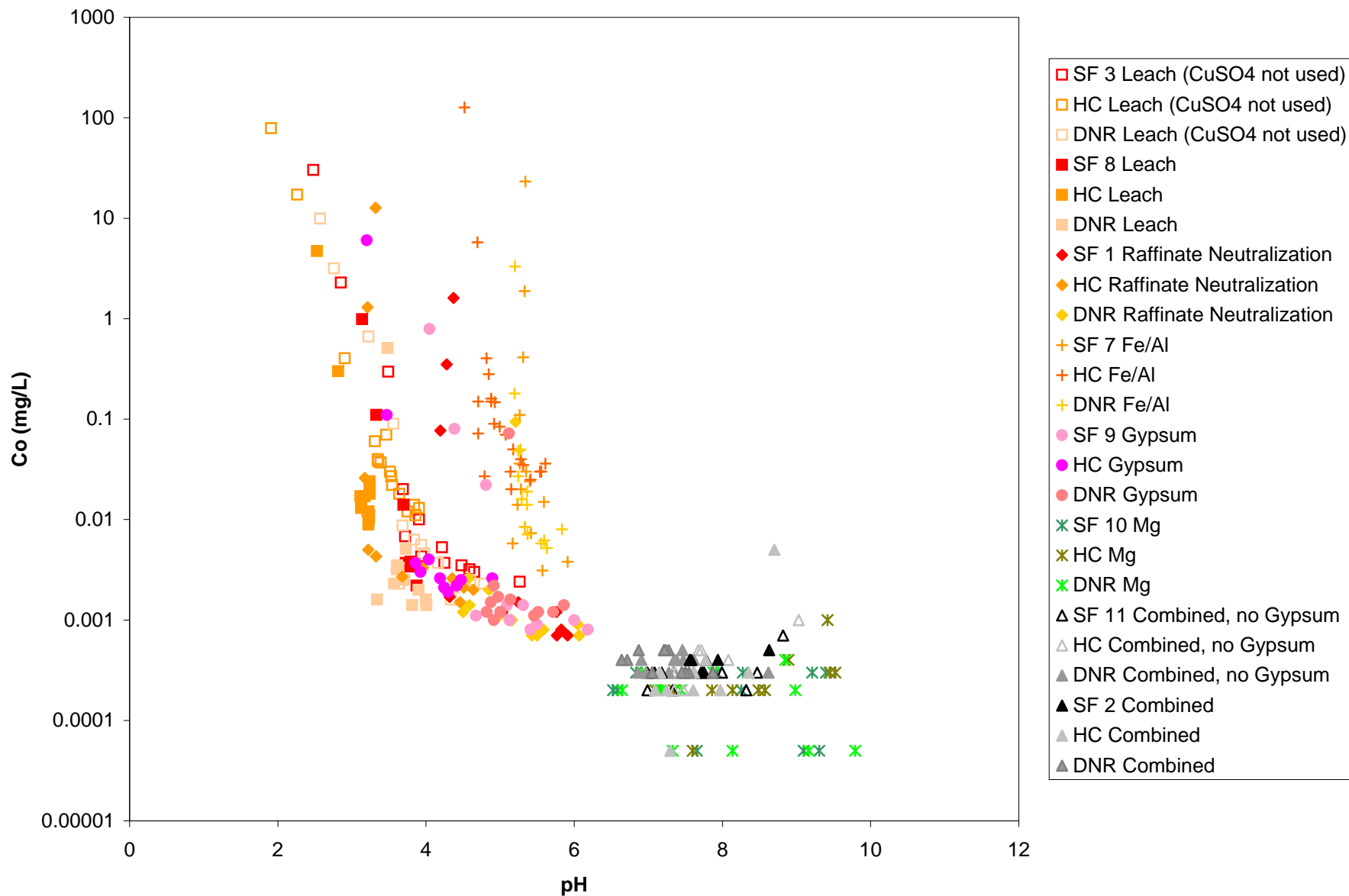
Graph D.5



M(pH)

Appendix D
Scatter Plots Showing Metal Concentrations Compared to pH

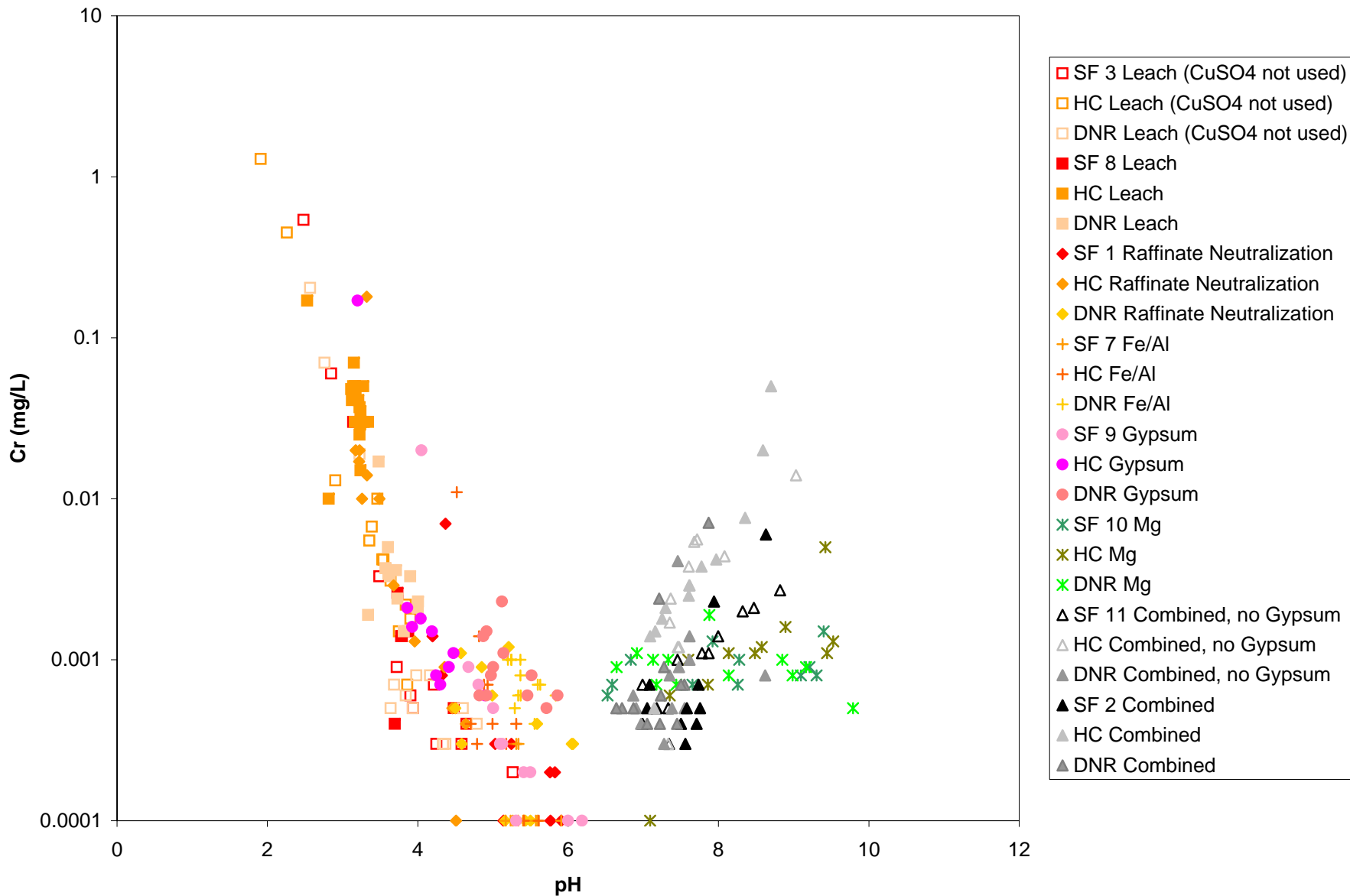
Graph D.6



M(pH)

Appendix D
Scatter Plots Showing Metal Concentrations Compared to pH

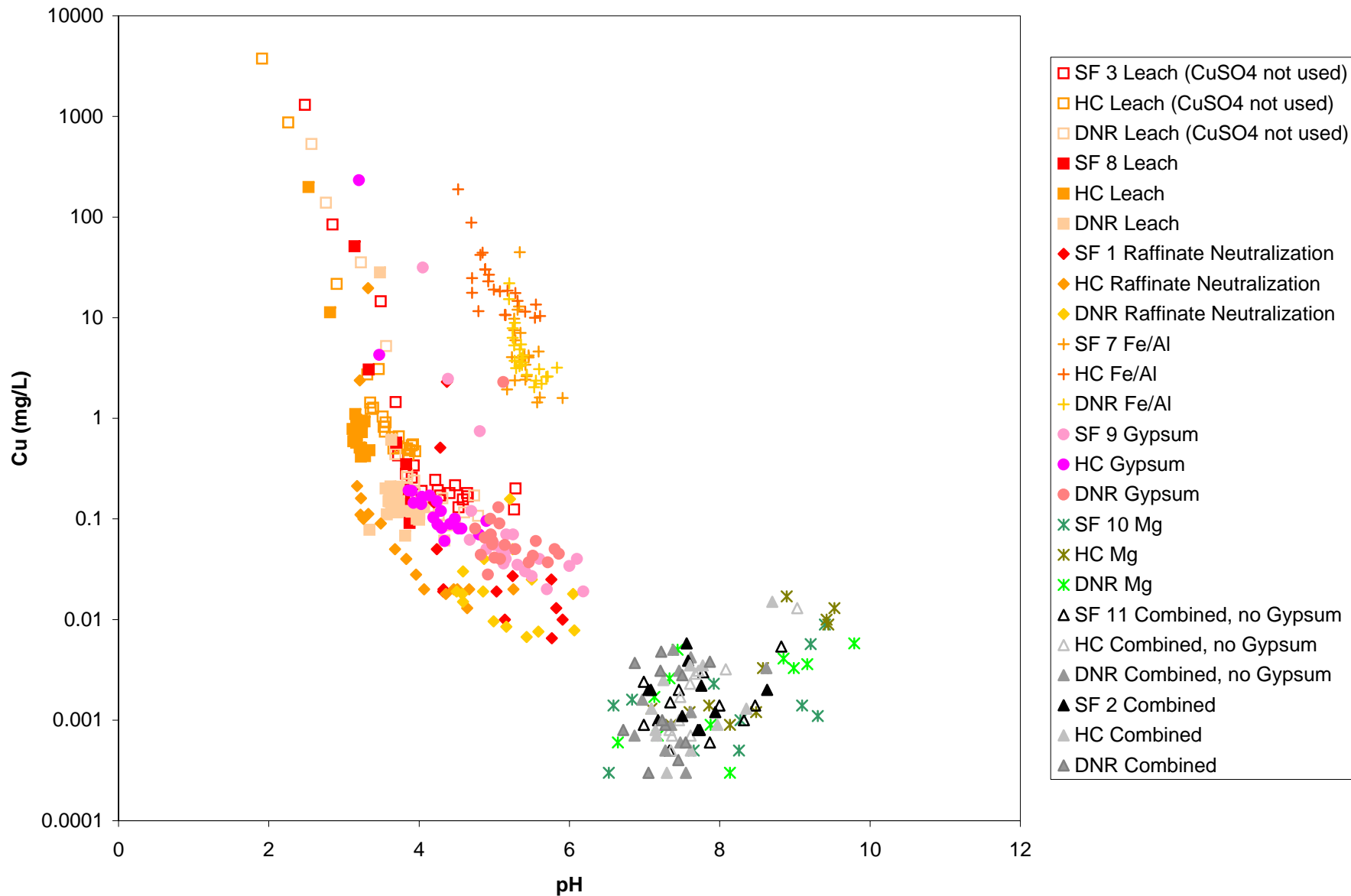
Graph D.7



M(pH)

Appendix D
Scatter Plots Showing Metal Concentrations Compared to pH

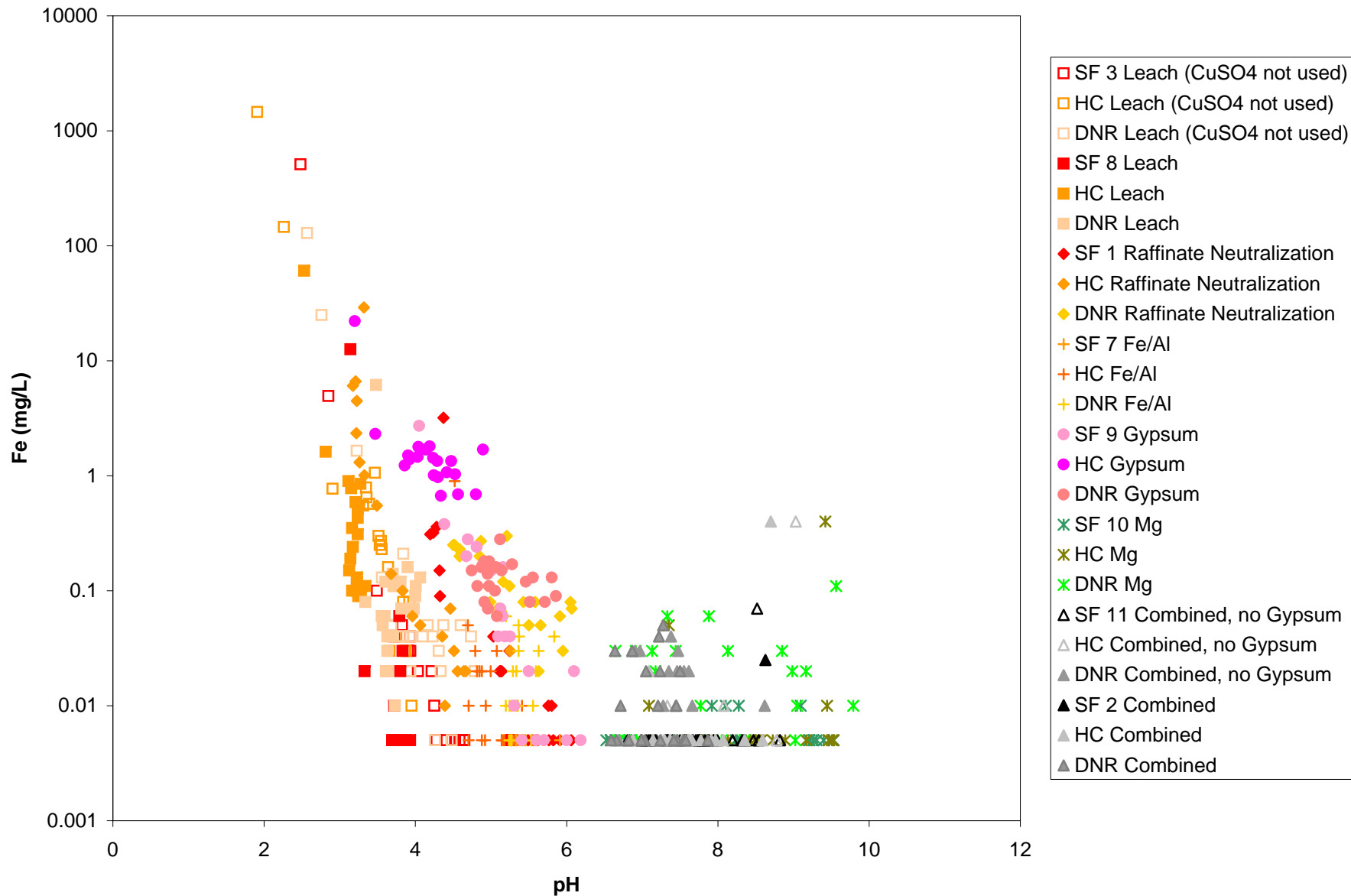
Graph D.8



M(pH)

Appendix D
Scatter Plots Showing Metal Concentrations Compared to pH

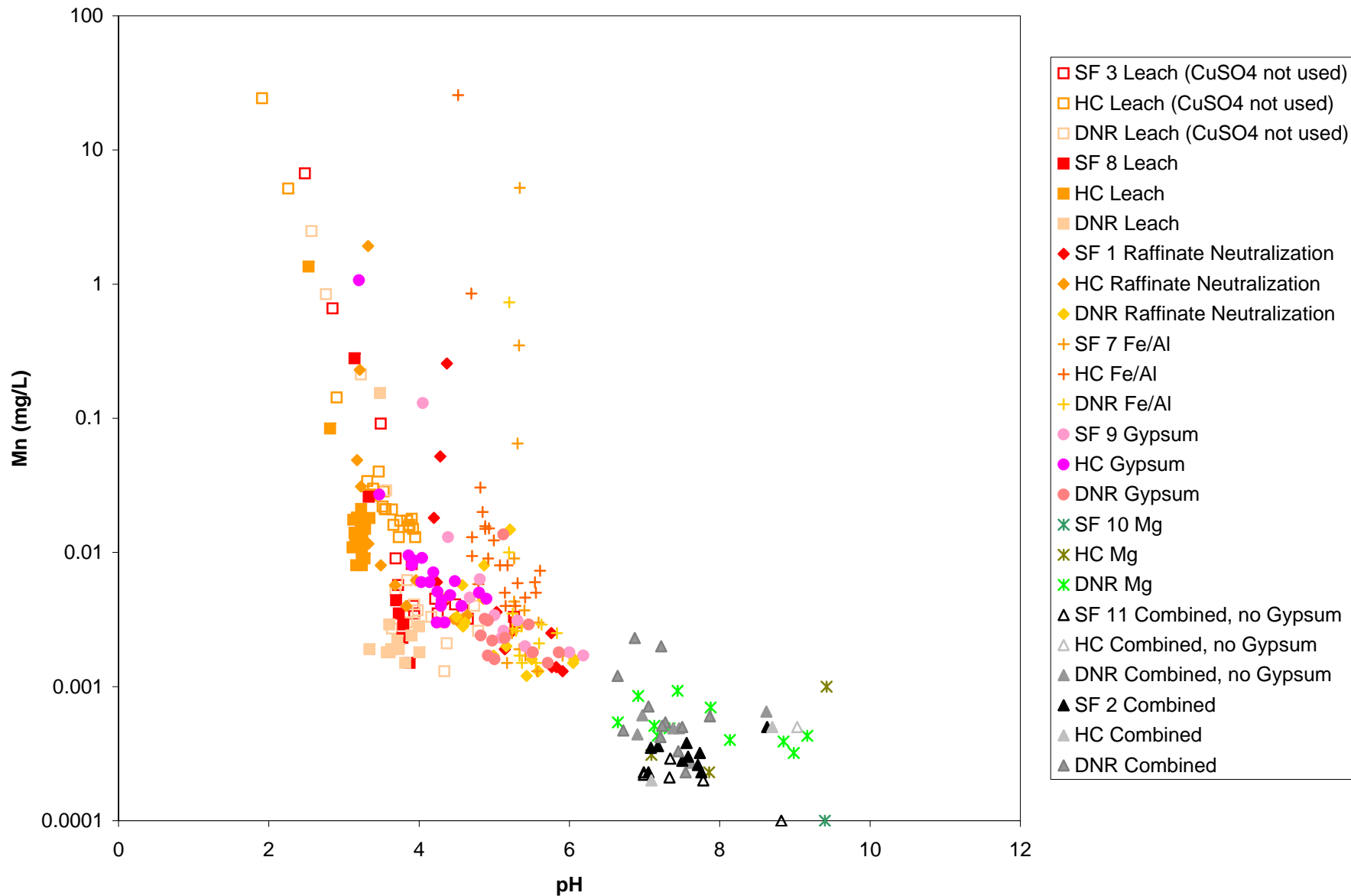
Graph D.9



M(pH)

Appendix D
Scatter Plots Showing Metal Concentrations Compared to pH

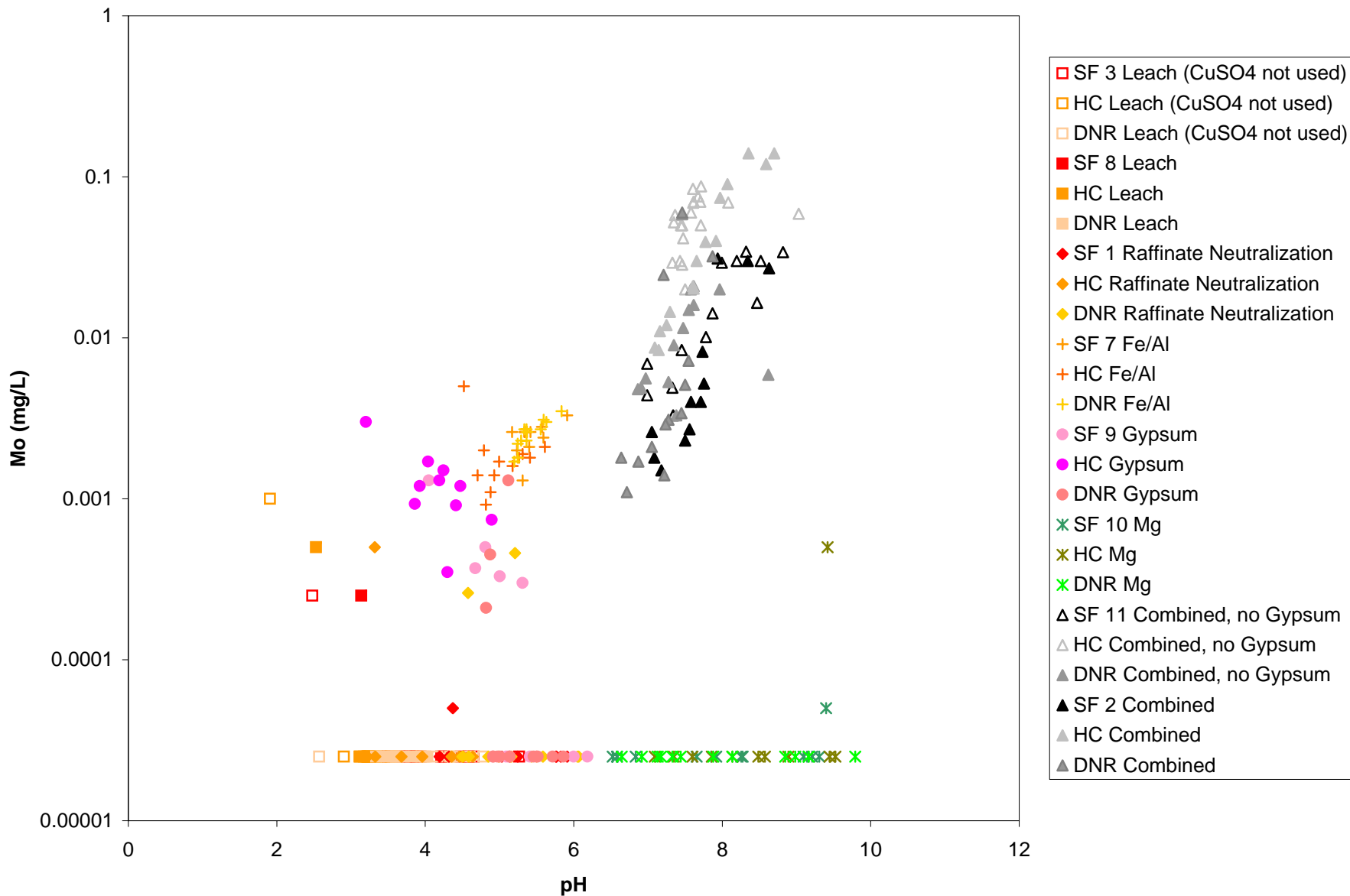
Graph D.10



M(pH)

Appendix D
Scatter Plots Showing Metal Concentrations Compared to pH

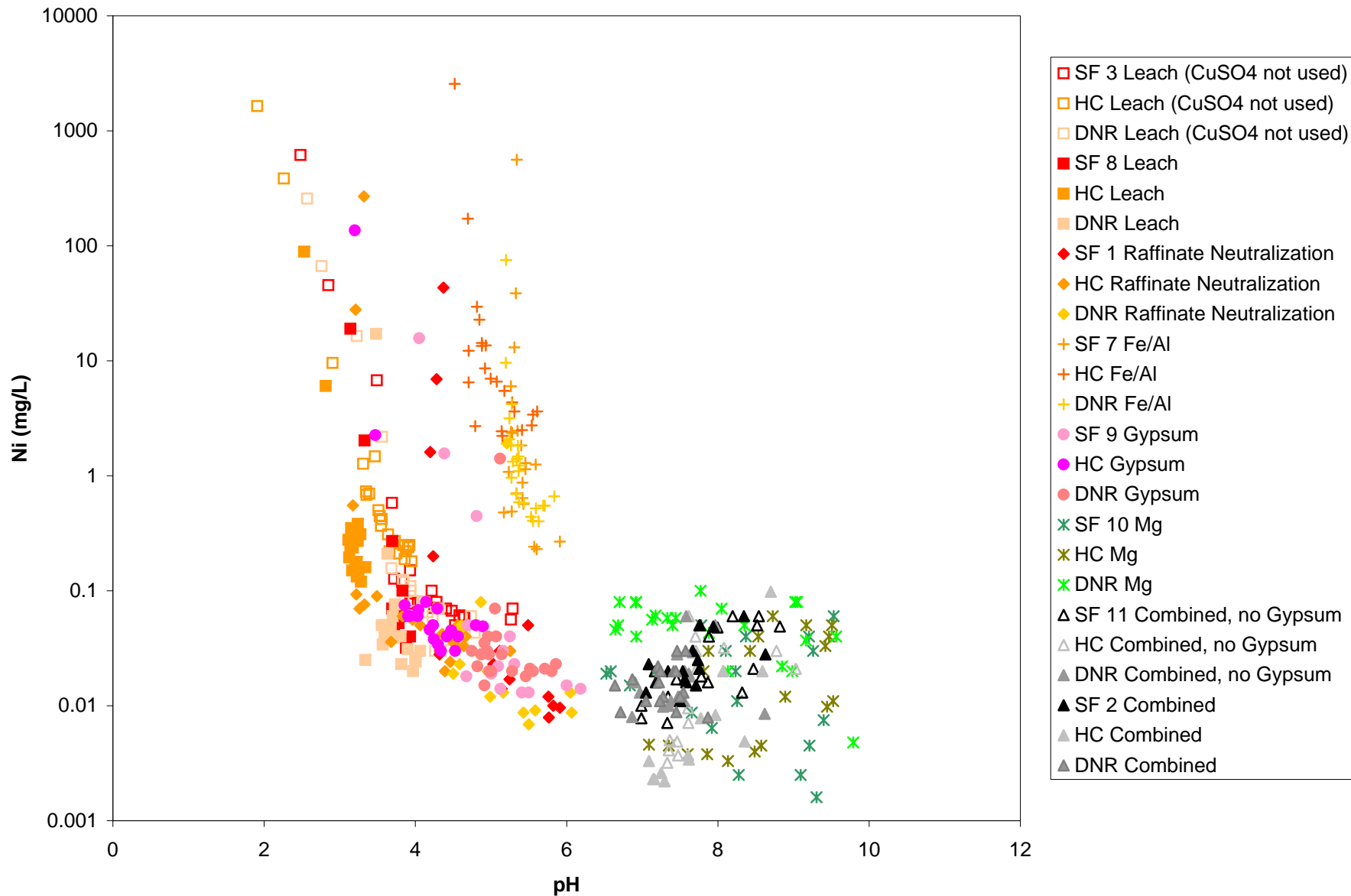
Graph D.11



M(pH)

Appendix D
Scatter Plots Showing Metal Concentrations Compared to pH

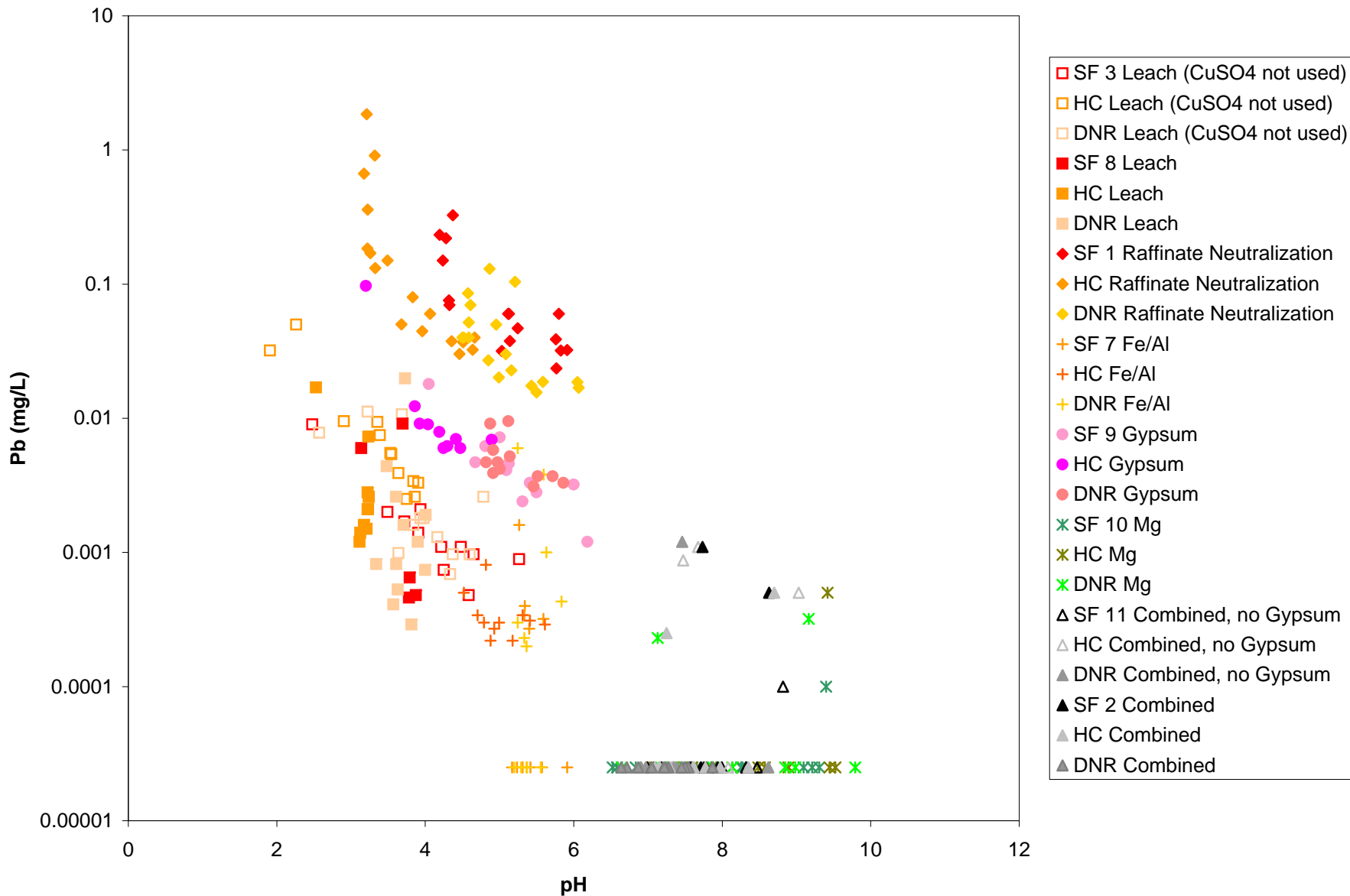
Graph D.12



M(pH)

Appendix D
Scatter Plots Showing Metal Concentrations Compared to pH

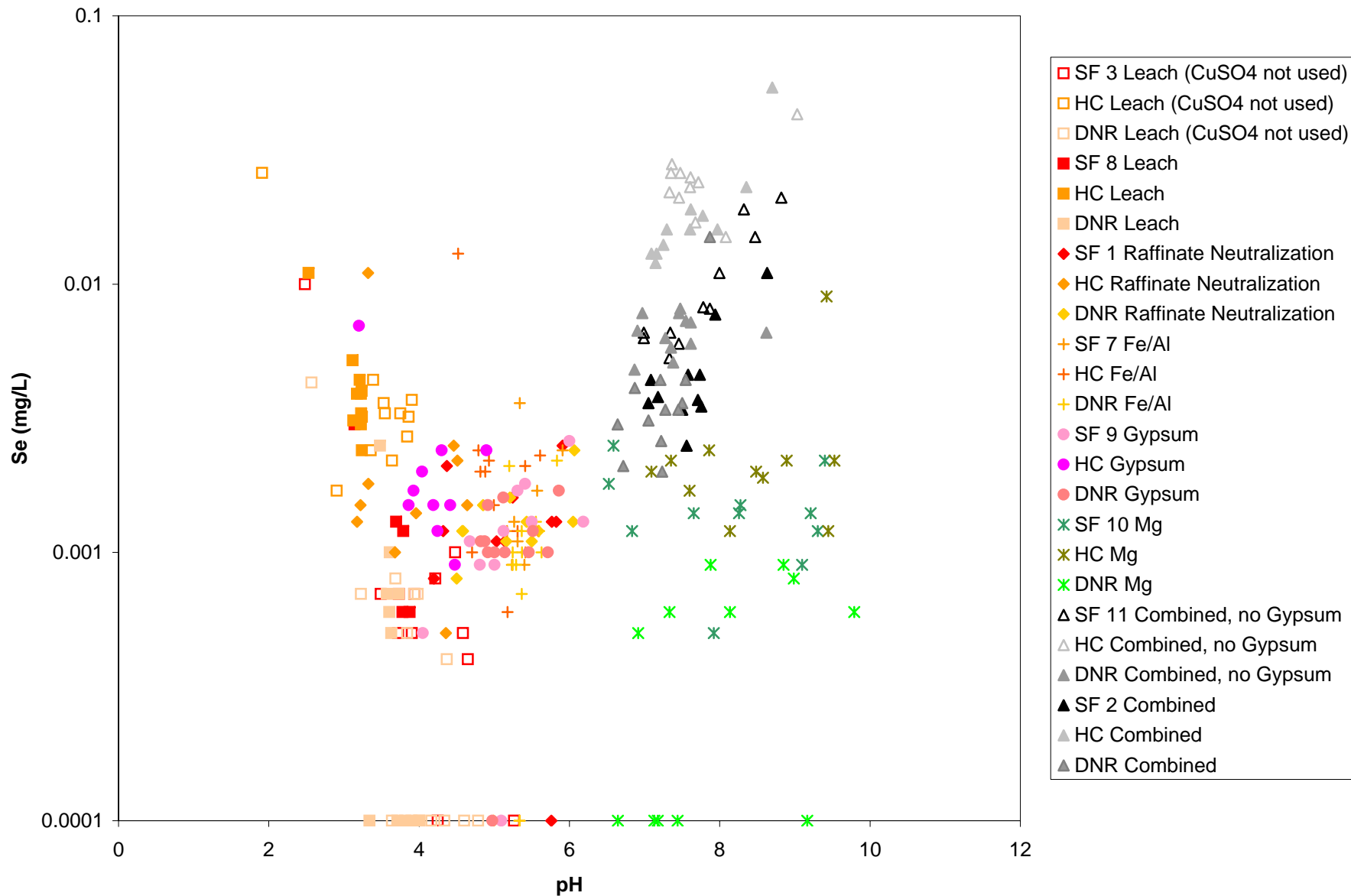
Graph D.13



M(pH)

Appendix D
Scatter Plots Showing Metal Concentrations Compared to pH

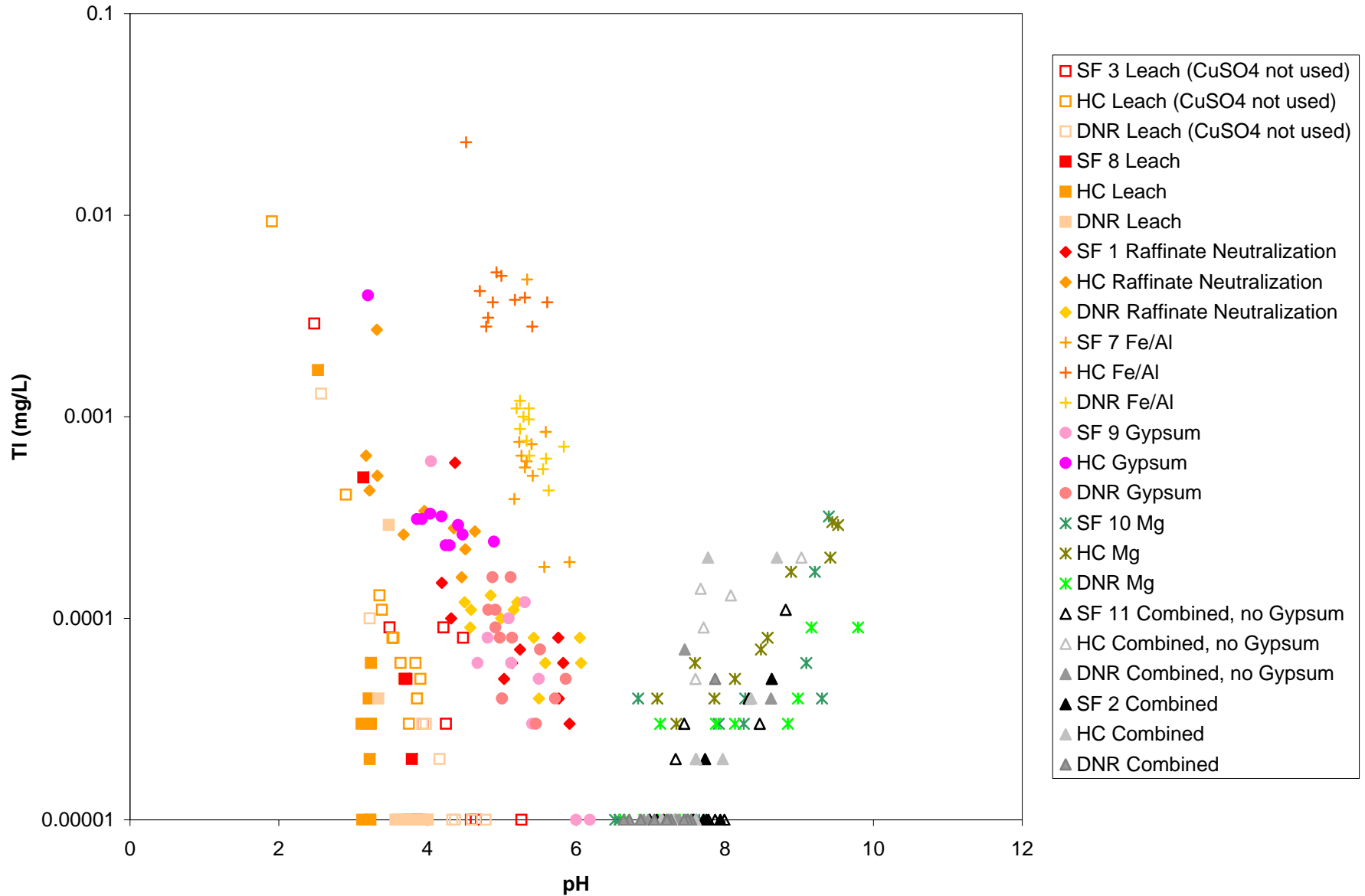
Graph D.14



M(pH)

Appendix D
Scatter Plots Showing Metal Concentrations Compared to pH

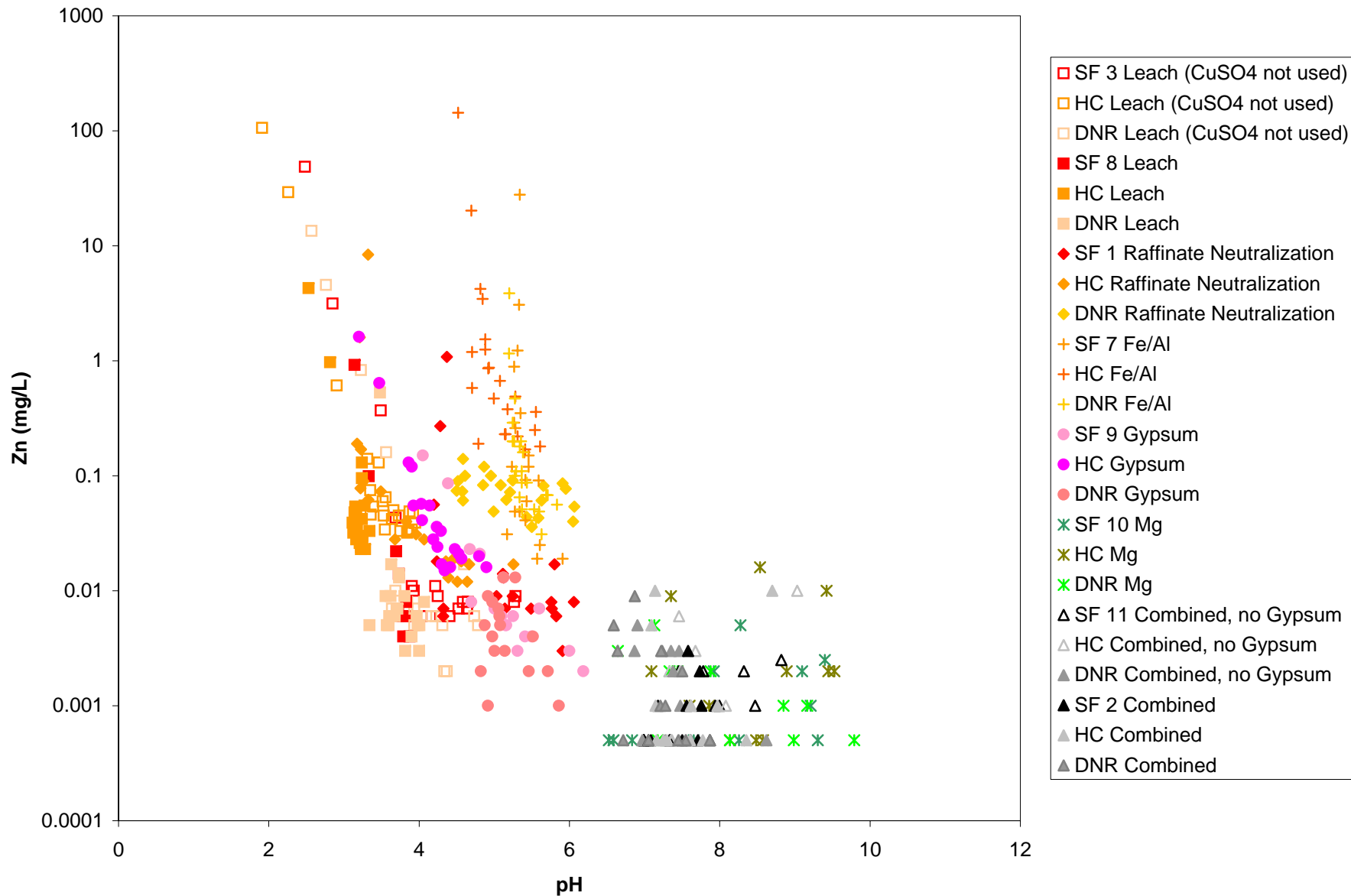
Graph D.15



M(pH)

Appendix D
Scatter Plots Showing Metal Concentrations Compared to pH

Graph D.16



M(pH)