

PARTICLE SIZE DISTRIBUTION STUDY OF POLYMETALLIX™ nZVI

1.0 EXECUTIVE SUMMARY

Lehigh University and The Whitman Companies, Inc. (Whitman) were retained by ARCADIS to develop particle size distributions (PSD) of four (4) lots of PolyMetallix™ nZVI prepared by the Polyflon Division of Crane Co. Following extensive analysis of the nZVI lots, our joint interpretation of the data are as follows. The majority (> 60%) of the nZVI evaluated for Samples #1, #2, and #3 were in the 50-100 nm diameter range which is generally consistent with the previous (August 2004) findings for an earlier PolyMetallix™ particle type. The nZVI in sample #4 were appreciably larger with the majority (> 70%) in the 125-300 nm range. This likely resulted from the fact that the Sample 4 nZVI was four months old at the time the PSD determination was made as compared to less than 2 weeks old for Samples 1-3. Thus, the degree of aggregation occurring in the older iron appears to be less reversible and/or amenable to size reduction. Consequently, based on these results, freshly manufactured nZVI in concert with physical size reduction and use of dispersant yielded the most favorable PSD characteristics insofar as subsurface mobility is concerned. However, this does not necessarily imply the nZVI will exhibit superior reactivity in the field. Additional investigations would be needed to develop this particular relationship.

2.0 INTRODUCTION

In April 2006, ARCADIS engaged Whitman and Lehigh University to perform a PSD study of four (4) lots of PolyMetallix™ nanoscale zero-valent iron (nZVI) prepared by the Polyflon division of Crane Co. The purpose of this study was to determine the PSD of the PolyMetallix™ nZVI by manual particle counting of images produced by Transmission Electron Microscopy (TEM). The images would be evaluated by highly trained personnel experienced in the handling and analysis of reactive iron nanoparticles.

In general, the lots were freshly prepared prior to shipment to Lehigh. It is not known whether the nZVI shipped was produced via differing or the same synthetic method(s). Limited non-proprietary manufacturing-related details were furnished to Whitman and Lehigh. This enabled an approximate and qualitative means of distinguishing the lots. The post-manufacturing details are as follows and are succinctly summarized in Table 1:

Sample #1

Approximately 40 grams of nZVI (dry weight basis) was treated via a physical mixing (i.e. grinding, size reduction) process following manufacturing as well as the addition of a dispersing agent. Details regarding the dispersant (i.e. non-ionic, anionic, or cationic) were not furnished. This sample was submitted to Lehigh within five days of manufacture.

Sample #2:

Approximately 40 grams of nZVI (dry weight basis) was treated via a physical mixing (i.e. grinding, size reduction) process following manufacturing. No dispersing agent was added to this sample. This sample was submitted to Lehigh within five days of manufacture.

Sample #3:

Approximately 40 grams of nZVI (dry weight basis) was not treated via the physical mixing (i.e. grinding, size reduction) process following preparation, but did include the addition of a dispersing agent. Details regarding the dispersant (i.e. non-ionic, anionic, or cationic) were not furnished. This sample was submitted to Lehigh within five days of manufacture.

Sample #4:

Approximately 40 grams of nZVI (dry weight basis) from a lot of nZVI manufactured four months before submittal to Lehigh formed the basis of Sample #4. However within five days of its submittal to Lehigh, this sample was treated via a physical mixing (i.e. grinding, size reduction) process following as well as the addition of a dispersing agent. Details regarding the nature of the dispersant (i.e. non-ionic, anionic, or cationic) were not furnished.

Table 1. Summary of Post-Manufacturing Treatment of Samples by Polyflon

SAMPLE #	DRY WT MASS, nZVI (g)	nZVI AGE¹	PHYSICAL SIZE REDUCTION²	USE OF DISPERSANT³
1	40	< 2 weeks	Yes	Yes
2	40	< 2 weeks	Yes	No
3	40	< 2 weeks	No	Yes
4	40	> 4 months	Yes	Yes

Notes:

- 1– Refers to the approximate age of nZVI prior to PSD determination at Lehigh
- 2– Post-manufacturing physical mixing (i.e. grinding) to generally accomplish size reduction.
- 3– Refers to the post-manufacturing addition of a dispersing agent to the nZVI matrix.

This report contains a summary of the analytical and data evaluation methodologies, a discussion of the salient results, and the raw data tables and PSD figures for each of the four (4) PolyMetallix™ iron samples. Comparisons to the PSD data from the August 2004 study entitled Physical Characterization of PolyMetallix™ Nanoscale Iron are also provided.

3.0 ANALYTICAL & DATA EVALUATION METHODOLOGIES

The PolyMetallix™ nZVI samples were received by Lehigh and analyzed without additional processing or modifications except where required by the method. As noted in Section 2 of this report, each sample contained 40 grams of nZVI on a dry weight basis suspended in matrices as follows: Sample #1: 250 milliliters (mL) aqueous solution with dispersing agent, Sample #2: 250 mL aqueous solution and ethanol, Sample #3: 500 mL aqueous solution with dispersing agent, and Sample #4: 950 mL aqueous solution with dispersing agent. The identity of the dispersing agent was not provided.

The general analytical protocol utilized was developed by Dr. Wei-xian Zhang at Lehigh University and was as follows. The samples were prepped for analysis by depositing 2-3 droplets of the dilute nZVI suspension onto a carbon film (Ernest Fullam, Latham, NY). The samples were then placed in a vacuum hood until the matrix was completely evaporated. The film containing the nZVI was then placed in a Philips EM 400T Transmission Electron Microscope (TEM, Philips Electronics Co., Eindhoven, The Netherlands) operating at 100 kilovolts (kV).

The actual particle size of the PolyMetallix™ nZVI clusters and aggregates was determined using Microsoft Photoshop according to the following procedure. The original TEM image contains a scale bar. The length of the scale bar is measured as a certain number of pixels. For the purposes of this illustration, assume that the scale bar is denoted as 100 nm and its length in pixels is denoted as A. Using Photoshop, the diameter of a representative number of nZVI clusters and aggregates can then be measured from the TEM. Let this diameter be equal to B pixels. Using Equation 1, the actual size of the nZVI cluster or aggregate can be calculated:

$$\text{nZVI particle size, nm} = (B * 100 \text{ nm})/A \quad [1]$$

For each PolyMetallix™ nZVI sample, twenty (20) images were obtained using a scaling range of approximately 50 nm to 1 micrometer, μm . This facilitated evaluation of a wide variety of nZVI clusters, aggregates, and assemblages. In general, all four samples of PolyMetallix™ nanoscale iron exhibited moderate to significant degrees of aggregation. This will be discussed in greater detail in Section 4.0 below.

4.0 FINDINGS AND RESULTS

The findings and results of the PolyMetallix™ nZVI PSD study are presented herein and include a general description of the particle morphology and aggregation (Section 4.1) as well as the PSD (Section 4.2). The raw data from the PSD determinations is included in Attachment 1. A summary of the salient PSD data is presented in Table 2. All of the PolyMetallix nZVI images are shown in Attachment 2 (PolyMetallix™ Image Library). The PSD graphs are shown in Figures 1 through 8. Figures 1, 3, 5, and 7 depict the number of particles counted as a function of diameter for each of the four (4) samples. Figures 2, 4, 6, and 8 show the distribution of the fraction of the total number of particles counted versus diameter.

The particle morphology is an important parameter as shape can strongly influence the subsurface mobility potential of nZVI, albeit probably to a lesser degree than size or surface charge. Based on empirical evidence from laboratory and field studies, spherical nZVI particle clusters exhibit better subsurface mobility properties than do more angular particles.

4.1 Particle Morphology and Aggregation Results

Sample #1 exhibited generally spherical particle clusters with a moderate proportion of particle clusters showing chain-like aggregation. Significant bud-like spherical nodules were observed on a number of the smaller aggregates (<50 nm). These are particularly evident in the images for Samples 1-3 and 1-4 (see Images Library in Attachment 2). These spherical nodules likely contribute significant additional surface area to the clusters which may correlate with enhanced reactivity. Some of the images exhibited the presence of foreign (i.e. non-nZVI) matter which may be residuals associated with the dispersing agent. This phenomenon is plainly shown in the image for Samples 1-5 and 1-10. A moderate amount of nZVI aggregation was observed in the images of Sample #1.

The TEM images for Sample #2 show more irregularly shaped clusters and aggregates of dense nZVI. A reasonable proportion of smaller clusters were observed but much less of the spherical nodules appear to be present. Although determination of specific surface area was not part of the scope of work, it is likely that that it is less for Sample #2 than for Sample #1 which could have intrinsic reactivity implications. Chain-like aggregates were observed in abundance as was the case with Sample #1. Moderate aggregation was observed in this Sample. Foreign matter possibly associated with the use of a dispersing agent was generally absent from these TEM images. As noted in Section 3.0, Sample #2 was not shipped with a dispersing agent in the matrix.

The TEM images for Sample #3 are similar to those of Sample #2. The nZVI particles appear as highly irregularly shaped clusters and aggregates. In general, the Sample #3 nZVI aggregates exhibit a more open structure which also would contribute to greater specific surface area. As mentioned previously, the specific surface area was not measured as part of this scope of work. The Sample #3 images show a moderate degree of particle aggregation and moderate evidence of foreign matter, likely residues from the dispersing agent used in the shipping matrix.

In general, the particle morphology of the Sample #4 TEM images indicates the presence of clusters and aggregates of spherical particles. However, some of the images depict nZVI with significant angularity (see images 4-1 and 4-6 in the Images Library, Attachment 2) which could be detrimental from a subsurface transport perspective. In light of the scale bar accompanying the images, these clusters and aggregates are appreciably larger than those for Samples #1 through #3, suggesting a larger extent of aggregation. Some evidence of the spherical bud-like nodules were also observed (images 4-4 and 4-15 in the Images Library, Attachment 2). Significant amounts of foreign matter were observed on most of the images, likely the residue of the dispersing agent used.

4.2 PSD Determination

The raw data for the PSD figures is included as Attachment 1 to this report. The actual PSDs are depicted in Figures 1-8. As noted previously, two plots are furnished for each sample. The first (Figures 1, 3, 5, and 7) show the distribution of the total particle count for each diameter size. The second graph depicts a normalized representation of the data wherein the fraction of the total particle count is shown as a function of diameter.

As provided in the raw data tabulation in Attachment 1, the total number of nZVI particles counted for each sample was as follows: #1 – 119, #2 – 181, #3 – 63, and #4 – 70. In general, the maximum, median, and minimum particle sizes for Samples #1 through #3 were reasonably similar. A summary of the PSD data for Samples 1-4 is provided in Table 2 below. Maximum particle sizes for Samples #1, #2, and #3 were 189, 252, and 164 nm, respectively. Median values were 66, 71, and 68.5 nm, respectively, for Samples #1 through #3 in ascending order. The minimum particle sizes for the three samples were very similar: 18.0, 18.7, and 13.3 nm respectively. However, the maximum, median, and minimum particle sizes for Sample #4 were 513, 187, and 38 nm, respectively.

Table 2. Summary of Polyflon PolyMetallic nZVI PSD Data

SAMPLE #	MEAN nZVI SIZE (nm)	MINIMUM nZVI SIZE (nm)	MAXIMUM nZVI SIZE (nm)
1	66.0	18.0	189.0
2	71.3	18.7	252.1
3	68.5	13.3	164.0
4	186.8	37.7	512.7

The PSD results for Samples 1 and 2 were also reasonably similar based on the mean and minimum particle size data. Moreover, the majority (>60%) of the particles counted were in the 50-100 nm range which is generally consistent with the findings from the August 2004 study performed on behalf of Crane Co. and Polyflon. Bell-shaped distribution curves were obtained for both Samples 1 and 2. However, the maximum particle size observed for Sample 2 was moderately larger than that for Sample 1, 252 nm versus 189 nm, which may be attributable to the lack of dispersing agent used in the post-manufacturing sample treatment process. However, this magnitude of difference will likely not contribute to a significant diminution of subsurface mobility potential.

As was the case for Samples 1 and 2, the majority (>60%) of the nZVI particles in Sample 3 were in the 50-100 nm size range. The PSD results for Samples 1 and 3, together with the post-manufacturing treatment information summarized in Table 1, suggest that the physical size reduction technique did not substantively affect the observed particle size insofar as the mean, minimum, and maximum parameters are concerned. However, the morphology of the PSD curves for Samples 1 and 3 was quite dissimilar, the latter exhibiting two maxima (approximately 50 and 85 nm, respectively). While the specific cause(s) for this are not known, it is possible that had physical size reduction been performed on Sample 3, the PSD results may have been similar to those from Sample 1. That is, larger nZVI aggregates in Sample 3 may have been broken apart by physical size reduction, yielding PSD data and curve similar to that for Sample 1.

The PSD for Sample 4, like those of Samples 1 and 2, was bell-shaped but the sizes were considerably larger. The majority (>70%) of nZVI particles counted were in the 125-300 nm size range, more than double the size observed in all other samples. An inspection of the data in Table 1 sheds light onto this apparent significant discrepancy. Specifically, although Sample 4 received physical size reduction and dispersant, it was manufactured four months prior to the PSD study and consequently was allowed to “age” much more than the other samples (Samples 1-3 were manufactured less than two weeks prior to the PSD study). This suggests that the degree of aggregation occurring in older stored nZVI is less reversible than in fresher iron samples. Moreover, the particle size reduction technique employed was not sufficient to result in the creation of significantly smaller iron particles. Moreover, despite the use of the dispersing agent, the Sample 4 iron was still substantially larger than the nZVI from Samples 1-3. This size differential is probably substantial enough to result in reduced subsurface mobility potential. However, the larger particle size of Sample 4 nZVI may not necessarily translate into lessened intrinsic reactivity.

5.0 CONCLUSIONS & RECOMMENDATIONS

Based on the analytical work of Lehigh University and our joint interpretation of the data, the majority (> 60%) of the nZVI evaluated for Samples #1, #2, and #3 were in the 50-100 nm diameter range which is generally consistent with the previous (August 2004) findings for an earlier PolyMetallix™ particle type. The nZVI in sample #4 were appreciably larger with the majority (> 70%) in the 125-300 nm range. This likely resulted from the fact that the Sample 4 nZVI was four months old at the time the PSD determination was made as compared to less than 2 weeks old for Samples 1-3. Thus, the degree of aggregation occurring in the older iron appears to be less reversible and/or amenable to size reduction. Consequently, based on these results, freshly manufactured nZVI in concert with physical size reduction and use of dispersant yielded the most favorable PSD characteristics insofar as subsurface mobility is concerned. However, this does not necessarily imply the nZVI will exhibit superior reactivity in the field. Additional investigations would be needed to develop this particular relationship.

While the PSD is an important quality assurance and quality control parameter, it alone is not a sufficient indicator of nZVI reactivity or efficacy in a given remediation scenario. It is important to emphasize that nZVI in general are highly reactive materials and, as such, their surface and intrinsic properties change rapidly over time from the time of manufacture. In addition to PSD, important physicochemical parameters include surface (or zeta) potential, isoelectric point, pH/ORP profile, etc. Moreover, short-term (i.e. <24 hours) standardized reactivity studies may be warranted to demonstrate efficacy for complex groundwater plumes, challenging geochemical environments, or source area applications with DNAPL. Finally, since little is presently known about the reactivity profile of nZVI over time in storage (i.e. between initial manufacturing and field utilization), Whitman and Lehigh recommend that a detailed characterization of the nZVI be performed to evaluate the rate of surface oxidization that occurs and identify the specific functional groups and valence states present. This knowledge would be an invaluable tool in helping to improve and optimize the reactivity of the nZVI surface and may lead to the development of better storage techniques for these highly reactive particles.

RAW ANALYTICAL DATA

Figure 1 - PSD: PolyMetallix #1

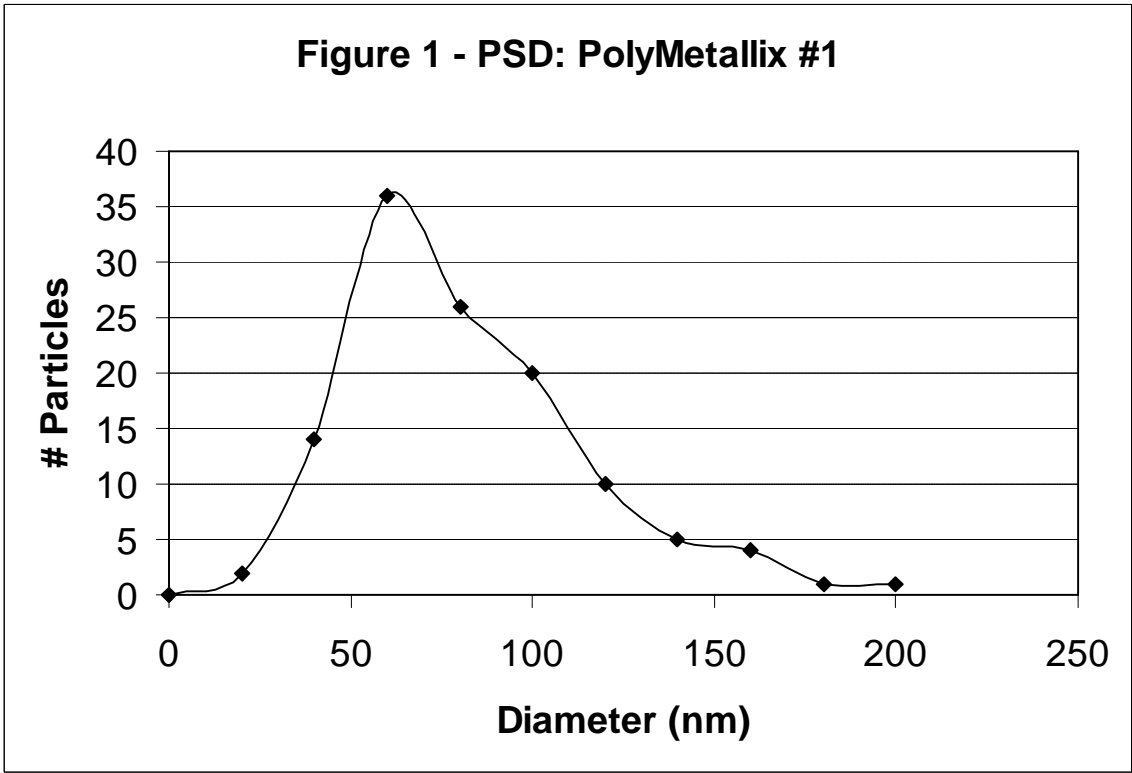
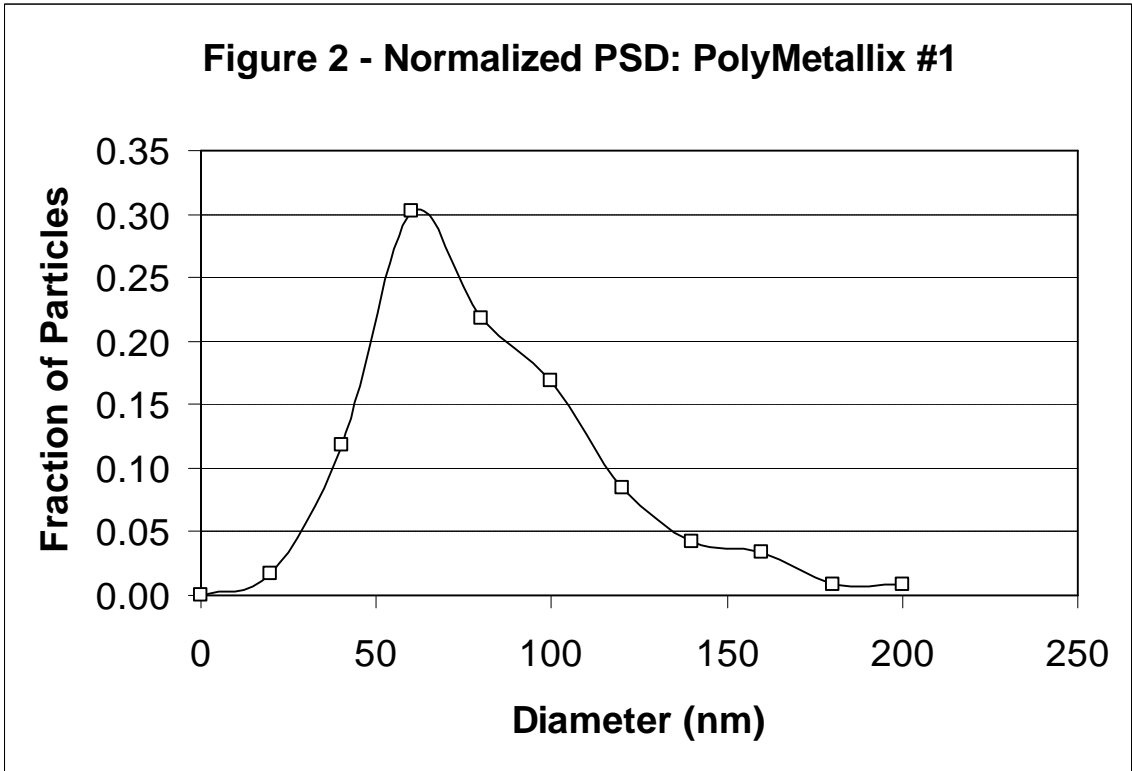


Figure 2 - Normalized PSD: PolyMetallix #1



II. Particle Size Maximum, Minimum, Median (Sample#1)

Maximum	189.04
Minimum	18.04
Median	66.00

III. Particle Size Distribution (See Graphs, Sample#1)

Diameter (nm)	Frequency	Normalized Frequency
0	0	0.00000
20	2	0.01681
40	14	0.11765
60	36	0.30252
80	26	0.21849
100	20	0.16807
120	10	0.08403
140	5	0.04202
160	4	0.03361
180	1	0.00840
200	1	0.00840
# of Particles	119	

Raw Data for Polyflon PolyMetallixtm Nanoscale nZVI Sample#1

Measured Diameter	Actual Diameter (nm)	Sorted Size (nm)	Measured Diameter	Actual Diameter (nm)	Sorted Size (nm)	Measured Diameter	Actual Diameter (nm)	Sorted Size (nm)
4.02	123.69	18.04	0.95	48.97	54.22	0.99	66.00	80.41
1.32	40.62	18.26	1.14	58.76	54.67	0.65	43.33	81.44
2.81	86.46	28.35	0.74	38.14	54.79	0.71	47.33	84.67
2.91	89.54	28.35	2.93	151.03	56.31	0.59	39.33	85.33
1.47	45.23	29.38	0.95	48.97	56.67	0.90	60.00	85.61
2.12	65.23	30.00	1.30	67.01	58.25	1.10	73.33	86.46
1.45	44.62	30.87	1.38	71.13	58.76	0.45	30.00	88.14
1.83	56.31	35.57	0.91	46.91	59.36	0.82	54.67	89.50
1.61	49.54	36.00	1.02	52.58	59.36	2.07	138.00	89.54
2.28	70.15	36.67	1.29	66.49	59.36	1.07	71.33	90.21
2.11	64.92	37.11	0.88	80.37	59.79	0.71	47.33	93.15
4.05	124.62	38.00	1.41	128.77	60.00	2.30	153.33	93.33
1.24	38.15	38.14	1.31	119.63	61.86	0.55	36.67	93.33
2.56	78.77	38.15	1.27	115.98	63.33	0.78	52.00	95.89
2.08	64.00	39.33	1.02	93.15	64.00	0.85	56.67	96.67
1.58	81.44	39.69	0.60	54.79	64.00	1.27	84.67	96.80
1.75	90.21	40.62	0.54	49.32	64.76	0.81	54.00	96.80
0.72	37.11	41.10	0.65	59.36	64.92	1.53	102.00	98.63
1.03	53.09	42.92	1.93	176.26	65.23	2.14	142.67	102.00
0.69	35.57	43.33	0.45	41.10	66.00	2.33	155.33	105.67
1.56	80.41	43.84	0.73	66.67	66.49	1.45	96.67	105.94
1.71	88.14	44.62	0.98	89.50	66.67	1.40	93.33	107.76
1.20	61.86	45.23	1.06	96.80	67.01	1.40	93.33	110.50
0.95	48.97	45.33	1.08	98.63	67.53	0.46	30.67	115.98
1.16	59.79	46.67	0.65	59.36	70.15	0.57	38.00	116.49
2.05	105.67	46.91	1.06	96.80	71.13	0.95	63.33	119.63
1.56	80.41	47.33	1.16	105.94	71.33	1.14	76.00	119.63
0.57	29.38	47.33	2.07	189.04	71.65	0.54	36.00	119.63
0.35	18.04	48.97	1.31	119.63	72.16	0.96	64.00	123.69
0.55	28.35	48.97	1.05	95.89	72.67	0.70	46.67	124.62
1.45	74.74	48.97	0.84	76.71	73.33	1.28	85.33	128.77
1.01	52.06	49.32	1.31	119.63	74.74	0.68	45.33	132.47
1.31	67.53	49.54	1.18	107.76	75.30	1.09	72.67	138.00
1.4	72.16	50.00	0.83	75.80	75.80	2.12	50.00	142.67
0.55	28.35	52.00	0.20	18.26	75.94	3.22	75.94	151.03
1.39	71.65	52.05	0.48	43.84	76.00	3.63	85.61	153.33
1.13	58.25	52.06	0.47	42.92	76.71	0.36	54.22	155.33
0.77	39.69	52.58	0.65	59.36	78.77	0.43	64.76	176.26
2.57	132.47	53.09	1.21	110.50	80.37	0.50	75.30	189.04
2.26	116.49	54.00	0.57	52.05	80.41			

Figure 3 - PSD: PolyMetallix #2

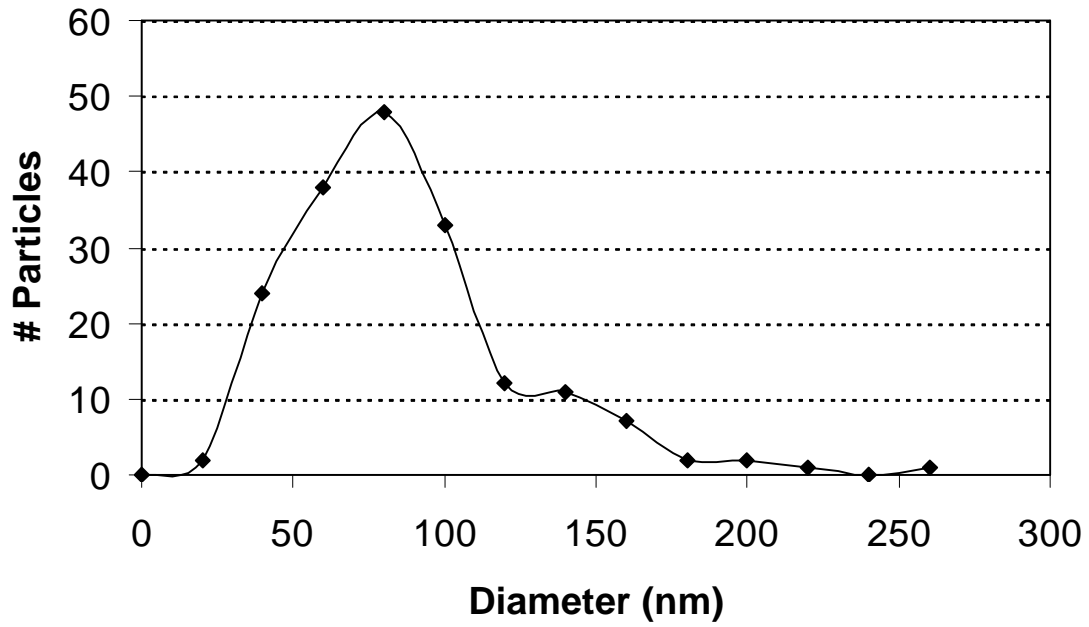
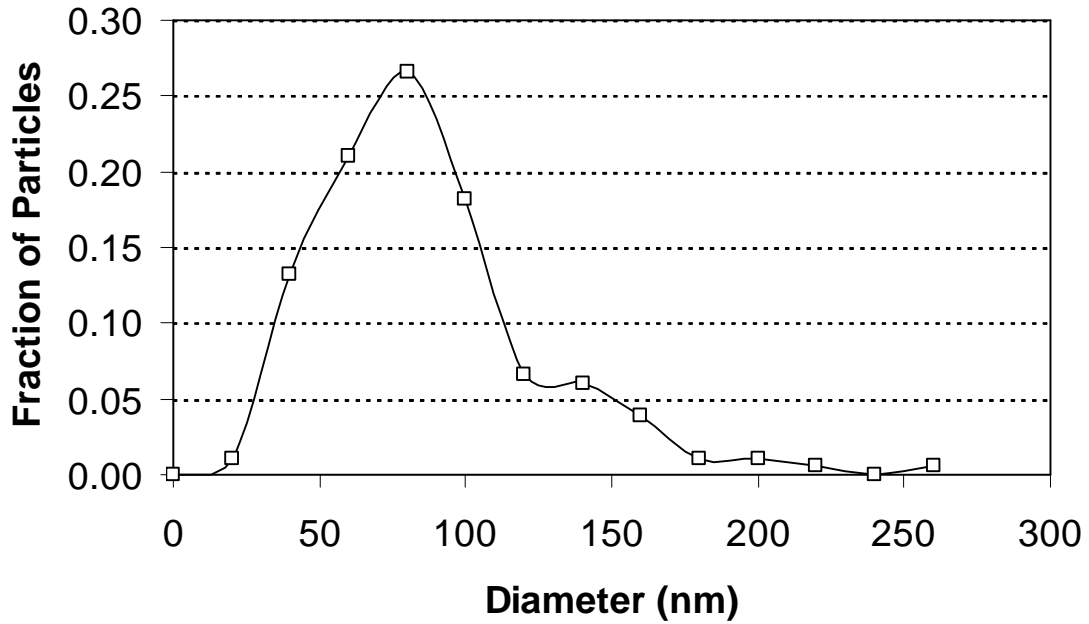


Figure 4 - Normalized PSD: PolyMetallix #2



II. Particle Size Maximum, Minimum, Median (Sample#2)

Maximum	252.09
Minimum	18.67
Median	71.33

III. Particle Size Distribution (See Graphs, Sample#2)

Diameter (nm)	Frequency	Normalized Frequency
0	0	0.00000
20	2	0.01105
40	24	0.13260
60	38	0.20994
80	48	0.26519
100	33	0.18232
120	12	0.06630
140	11	0.06077
160	7	0.03867
180	2	0.01105
200	2	0.01105
220	1	0.00552
240	0	0.00000
260	1	0.00552
# of Particles	181	

Raw Data for Polyflon PolyMetallixtm Nanoscale nZVI Sample#2

Meas. Diam.	Act. Diam (nm)	Sorted Size (nm)	Meas. Diam.	Act. Diam (nm)	Sorted Size (nm)	Meas. Diam.	Act. Diam (nm)	Sorted Size (nm)	Meas. Diam.	Act. Diam (nm)	Sorted Size (nm)
2.71	252.09	18.67	0.65	60.47	51.16	1.09	72.67	71.33	1.03	68.67	95.33
1.96	182.33	18.93	0.96	89.30	51.55	0.72	48.00	71.63	1.00	66.67	96.30
1.29	120.00	23.33	0.68	63.26	52.67	1.95	130.00	71.63	1.15	76.67	96.59
1.56	145.12	23.71	1.04	96.74	52.67	0.88	58.67	72.56	1.31	87.33	96.67
0.61	56.74	24.62	1.22	113.49	54.67	0.63	42.00	72.67	2.38	158.67	96.74
0.85	79.07	26.00	1.06	98.60	54.67	1.03	68.67	72.67	2.67	82.15	97.94
0.43	40.00	28.00	1.50	139.53	55.33	1.09	72.76	72.67	2.75	84.62	98.60
0.55	51.16	29.77	1.82	121.33	55.67	1.24	82.67	73.33	0.80	24.62	98.67
0.52	48.37	30.00	1.01	67.33	55.81	1.34	89.33	73.33	1.83	75.31	100.00
0.61	56.74	31.33	1.04	69.33	56.67	1.07	71.33	74.42	2.34	96.30	100.67
0.60	55.81	31.44	1.45	96.67	56.74	1.07	71.33	74.67	0.82	33.74	102.00
0.65	60.47	32.67	1.98	132.00	56.74	1.66	110.67	75.31	2.38	97.94	102.67
1.67	155.35	32.67	0.96	64.00	57.61	0.75	50.00	75.35	1.41	58.02	103.33
1.23	114.42	33.74	0.88	58.67	58.02	0.79	52.67	75.35	0.46	18.93	104.67
1.63	151.63	34.00	0.82	54.67	58.67	1.10	73.33	76.67	0.90	37.04	107.73
1.66	154.42	35.05	0.69	46.00	58.67	0.82	54.67	76.67	0.90	37.04	110.67
1.99	185.12	35.33	0.79	52.67	59.33	0.65	43.33	77.33	1.90	78.19	113.49
0.80	74.42	36.67	0.68	45.33	60.00	0.51	34.00	78.19	1.40	57.61	114.42
1.41	131.16	37.04	2.53	168.67	60.47	1.34	89.33	78.67	1.67	68.72	119.07
1.01	93.95	37.04	3.18	212.00	60.47	1.55	103.33	79.01	1.66	68.31	119.32
0.85	79.07	37.63	0.92	61.33	60.67	0.49	32.67	79.07	1.51	62.14	120.00
1.44	133.95	38.00	0.89	59.33	61.33	0.65	43.33	80.00	2.36	121.65	121.33
0.86	80.00	38.00	1.10	73.33	61.34	0.47	31.33	80.93	0.61	31.44	121.65
1.34	124.65	38.14	1.31	87.33	62.14	0.55	36.67	82.00	2.09	107.73	124.65
1.55	144.19	38.67	1.15	76.67	63.26	0.72	48.00	82.15	1.26	64.95	128.37
0.77	71.63	40.00	0.96	64.00	63.26	1.36	90.67	82.67	1.67	86.08	130.00
0.51	47.44	42.00	1.03	68.67	64.00	0.76	50.67	83.33	1.19	61.34	131.16
0.96	89.30	43.33	0.57	38.00	64.00	1.16	77.33	84.09	1.00	51.55	132.00
0.93	86.51	43.33	0.91	60.67	64.95	2.01	134.00	84.62	1.33	68.56	133.95
0.93	86.51	43.72	1.09	72.67	66.67	1.33	88.67	86.08	0.87	44.85	134.00
0.81	75.35	44.67	0.45	30.00	66.67	1.41	94.00	86.51	0.73	37.63	137.50
0.75	69.77	44.85	0.28	18.67	66.67	2.45	163.33	86.51	1.36	70.10	139.53
0.49	45.58	44.85	0.39	26.00	67.33	0.83	55.33	86.61	0.46	23.71	144.19
0.32	29.77	45.33	0.42	28.00	68.31	1.48	98.67	87.33	0.74	38.14	145.12
1.38	128.37	45.58	0.35	23.33	68.56	1.54	102.67	87.33	0.68	35.05	146.05
0.77	71.63	46.00	0.85	56.67	68.67	1.00	66.67	88.67	1.57	80.93	151.63
0.81	75.35	47.44	0.67	44.67	68.67	0.53	35.33	89.30	0.87	44.85	154.42
1.28	119.07	48.00	0.75	50.00	68.67	1.53	102.00	89.30	1.80	92.78	155.35
0.78	72.56	48.00	0.90	60.00	68.72	1.23	82.00	89.33	1.08	55.67	158.67
0.54	50.23	48.37	0.58	38.67	69.33	1.25	83.33	89.33	0.99	51.03	163.33
0.99	92.09	50.00	0.57	38.00	69.77	1.00	66.67	90.67	1.21	137.50	168.67
0.68	63.26	50.00	1.12	74.67	70.10	1.07	71.33	92.09	0.85	96.59	182.33
1.57	146.05	50.23	1.18	78.67	71.33	1.50	100.00	92.78	0.74	84.09	185.12
0.55	51.16	50.67	1.57	104.67	71.33	1.43	95.33	93.95	0.83	94.32	212.00
0.93	86.51	51.03	1.09	72.67	71.33	1.51	100.67	94.00	0.83	119.32	252.09
0.47	43.72	51.16	0.72	48.00	71.63	0.49	32.67	94.32			

Figure 5 - PSD: PolyMetallix #3

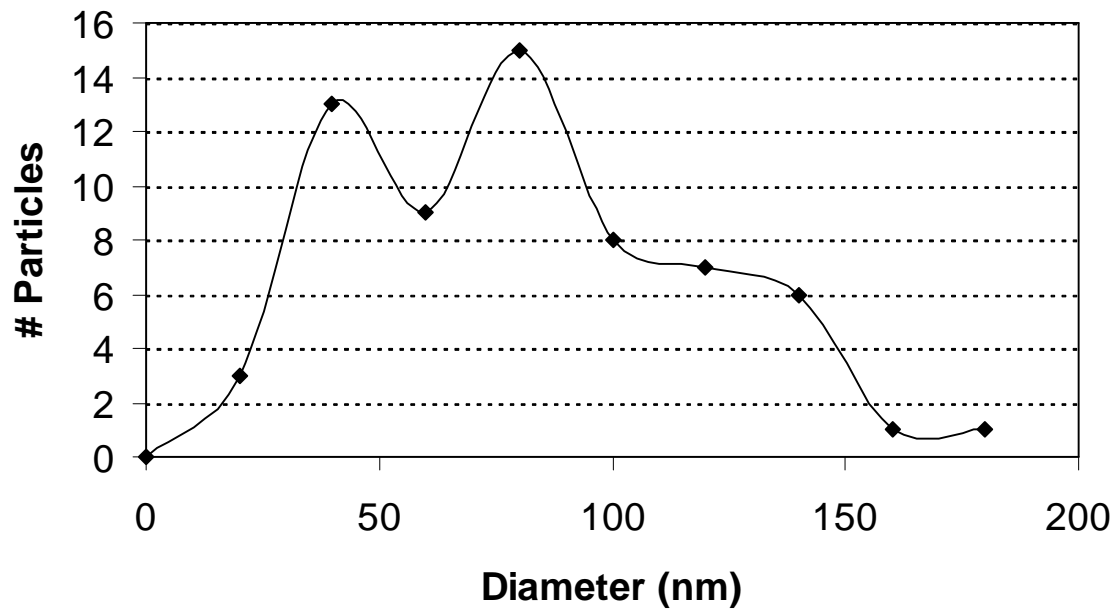
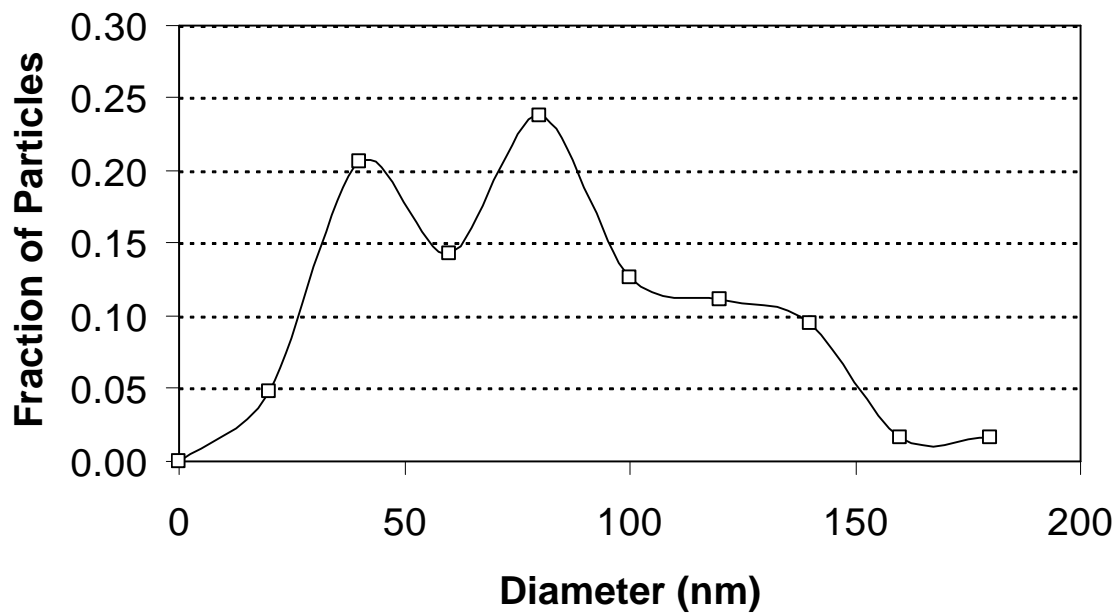


Figure 6 - Normalized PSD: PolyMetallix #3



II. Particle Size Maximum, Minimum, Median (Sample#3)

Maximum	164.00
Minimum	13.30
Median	68.49

III. Particle Size Distribution (See Graphs, Sample#3)

Diameter (nm)	Frequency	Normalized Frequency
0	0	0.00000
20	3	0.04762
40	13	0.20635
60	9	0.14286
80	15	0.23810
100	8	0.12698
120	7	0.11111
140	6	0.09524
160	1	0.01587
180	1	0.01587
# of Particles	63	

Raw Data for Polyflon PolyMetallix™ Nanoscale nZVI Sample#3

Meas. Diam.	Act. Diam (nm)	Sorted Size (nm)	Meas. Diam.	Act. Diam (nm)	Sorted Size (nm)	Meas. Diam.	Act. Diam (nm)	Sorted Size (nm)
0.85	77.63	13.33	1.13	103.20	54.67	2.14	65.85	89.50
1.29	117.81	18.33	0.75	68.49	54.79	1.82	56.00	92.31
1.05	95.89	19.00	0.78	71.23	55.71	1.62	49.85	95.89
1.39	126.94	21.33	0.85	77.63	56.00	3.25	100.00	97.72
1.39	126.94	23.33	0.98	89.50	61.85	3.00	92.31	99.54
1.62	147.95	23.67	0.86	78.54	63.93	3.33	102.46	100.00
1.16	105.94	25.00	1.14	104.11	64.84	1.02	34.00	100.46
1.80	164.38	26.48	0.70	63.93	64.84	1.03	34.33	102.46
0.75	68.49	27.00	0.47	42.92	64.84	0.71	23.67	103.20
0.71	64.84	28.67	1.10	100.46	65.85	1.64	54.67	104.11
0.29	26.48	30.33	0.40	36.53	68.49	1.20	40.00	105.94
0.71	64.84	34.00	0.60	54.79	68.49	1.20	19.00	114.16
1.50	136.99	34.33	1.50	136.99	68.49	0.57	30.33	117.81
1.41	128.77	36.53	1.25	114.16	71.23	0.91	28.67	120.55
0.50	45.66	38.00	1.09	99.54	73.06	0.86	25.00	126.94
1.07	97.72	40.00	0.75	68.49	77.63	0.75	27.00	126.94
1.32	120.55	41.10	0.86	78.54	77.63	0.81	23.33	128.77
0.80	73.06	42.92	0.97	88.58	78.54	0.70	18.33	136.99
0.49	44.75	44.75	0.71	64.84	78.54	0.55	21.33	136.99
0.61	55.71	45.66	0.45	41.10	88.58	0.64	13.33	147.95
0.97	88.58	49.85	2.01	61.85	88.58	0.40	38.00	164.38

Figure #7 - PSD: PolyMetallix #4

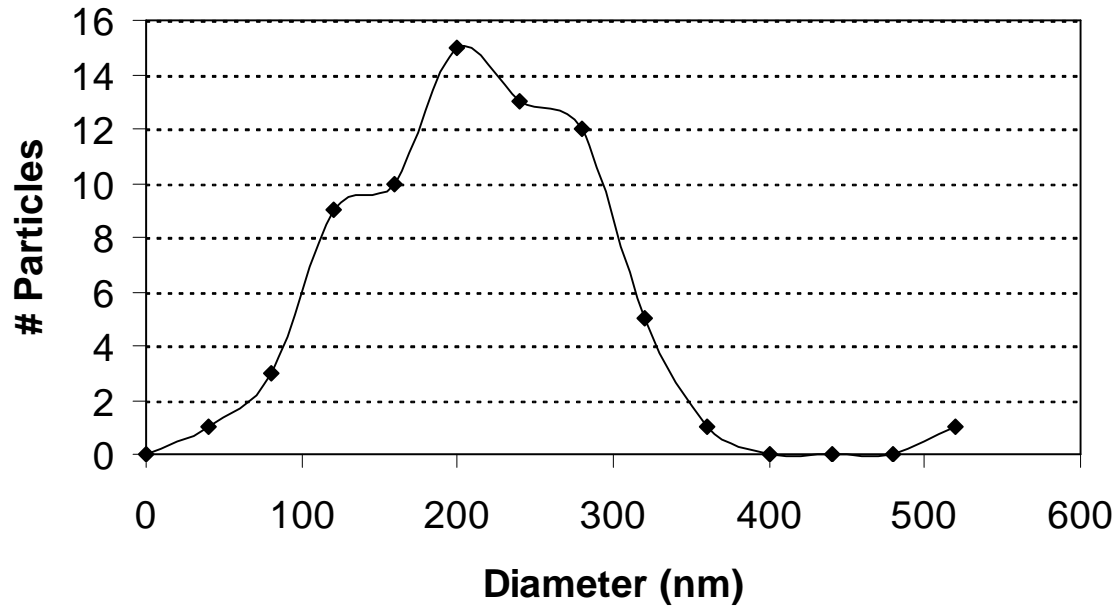
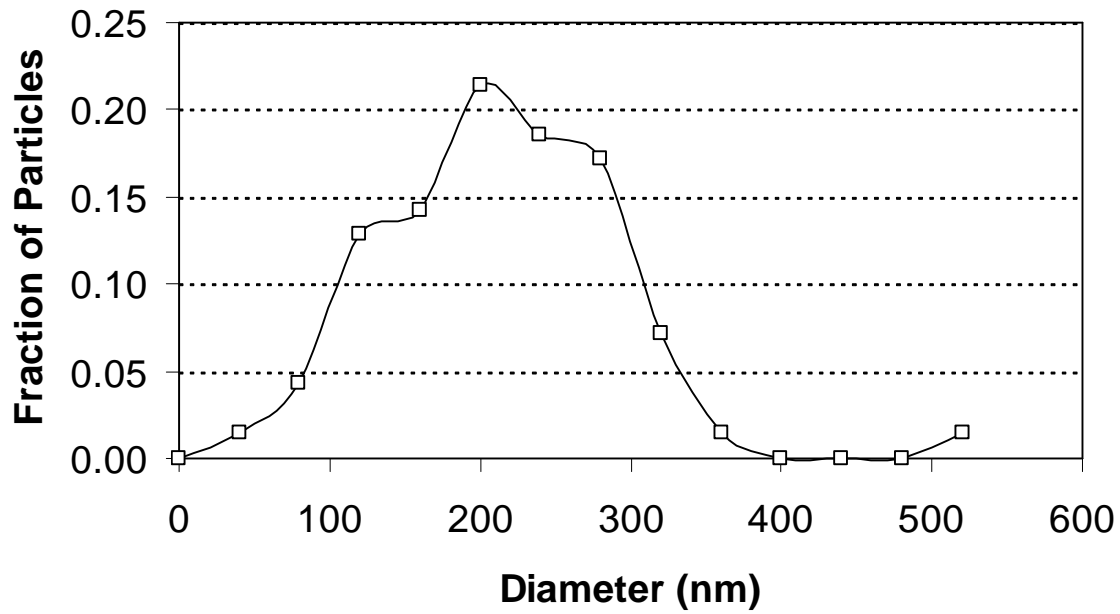


Figure #8 - Normalized PSD: PolyMetallix #4



II. Particle Size Maximum, Minimum, Median (Sample#4)

Maximum	512.70
Minimum	37.30
Median	186.75

III. Particle Size Distribution (See Graphs, Sample#4)

Diameter (nm)	Frequency	Normalized Frequency
0	0	0.00000
40	1	0.01429
80	3	0.04286
120	9	0.12857
160	10	0.14286
200	15	0.21429
240	13	0.18571
280	12	0.17143
320	5	0.07143
360	1	0.01429
400	0	0.00000
440	0	0.00000
480	0	0.00000
520	1	0.01429
# of Particles	70	

Raw Data for Polyflon PolyMetallixtm Nanoscale nZVI Sample#4

Meas. Diam.	Act. Diam (nm)	Sorted Size (nm)	Meas. Diam.	Act. Diam (nm)	Sorted Size (nm)	Meas. Diam.	Act. Diam (nm)	Sorted Size (nm)
2.82	512.73	37.65	1.34	201.81	165.66	1.12	168.67	220.00
1.41	256.36	58.73	0.83	125.00	167.27	1.23	185.24	232.73
1.50	272.73	60.24	1.26	189.76	168.67	0.58	87.35	236.45
1.55	281.82	79.82	1.40	210.84	170.91	0.75	112.95	243.64
0.90	163.64	87.35	1.25	188.25	172.73	0.64	96.39	243.98
0.82	149.09	90.91	0.75	112.95	173.19	1.62	243.98	247.27
2.03	369.09	96.39	1.82	274.10	180.72	1.33	200.30	252.73
1.53	278.18	100.90	0.94	141.57	182.23	1.78	268.07	256.36
1.20	218.18	102.41	1.39	209.34	183.73	1.22	183.73	256.36
1.34	243.64	112.95	1.39	209.34	185.24	1.57	236.45	268.07
1.57	285.45	112.95	0.98	147.59	185.24	1.42	213.86	272.73
1.39	252.73	112.95	1.23	185.24	188.25	1.84	277.11	274.10
1.36	247.27	117.47	0.85	128.01	189.76	1.82	274.10	274.10
1.69	307.27	121.99	0.67	100.90	191.27	1.21	182.23	277.11
0.95	172.73	125.00	0.81	121.99	200.30	1.89	284.64	278.18
0.94	170.91	128.01	0.90	135.54	201.81	1.91	287.65	281.82
1.21	220.00	128.01	0.78	117.47	203.31	0.53	79.82	284.64
0.71	129.09	129.09	0.68	102.41	204.82	0.40	60.24	285.45
0.81	147.27	135.54	1.20	180.72	207.83	0.75	112.95	287.65
1.28	232.73	141.57	1.36	204.82	209.34	0.25	37.65	307.27
1.41	256.36	147.27	1.35	203.31	209.34	0.39	58.73	369.09
0.92	167.27	147.59	1.15	173.19	210.84	0.85	128.01	512.73
0.50	90.91	149.09	1.27	191.27	213.86			
1.38	207.83	163.64	1.10	165.66	218.18			