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Spectrometric Analysis for Pulse Jet Mixer Testing

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LIST OF ACRONYMS and ABBREVIATIONS

abs	**absorbance**
ADS	**Analytical Development Section**
CRV	**Concentrate Receipt Vessel**
DOE	**Department of Energy**
EDL	**Engineering Development Lab**
g	**grams**
nm	**nanometer**
PJM	**pulse jet mixer**
PNNL	**Pacific Northwest National Laboratory**
RPP-WTP	**Hanford River Protect Program- Waste Treatment Program**
SRS	**Savannah River Site (Aiken, SC)**
UV-Vis	**ultraviolet-visible**

ABSTRACT

The Analytical Development Section (ADS) was tasked with providing support for a Hanford River Protection Program-Waste Treatment Program (RPP-WTP) project test involving absorption analysis for non-Newtonian pulse jet mixer testing for small scale (PJM) and prototype (CRV) tanks with sparging. Tanks filled with clay were mixed with various amounts of powdered dye as a tracer. The objective of the entire project was to determine the best mixing protocol (nozzle velocity, number of spargers used, total air flow, etc.) by determining the percent mixed volume through the use of an ultraviolet-visible (UV-Vis) spectrometer. The dye concentration within the sample could be correlated to the volume fraction mixed in the tank. Samples were received in vials, a series of dilutions were generated from the clay, allowed to equilibrate, then centrifuged and siphoned for the supernate liquid to analyze by absorption spectroscopy. Equilibration of the samples and thorough mixing of the samples were a continuous issue with dilution curves being difficult to obtain. Despite these technical issues, useful data was obtained for evaluation of various mix conditions.

1.0 BACKGROUND

During pulse jet mixer (PJM) testing of Hanford's Concentrate Receipt Vessel (CRV), it is difficult to visually detect the mixed volume within the tank. A spectrophotometric method was developed where a dye is injected into the mixed zone and at different operating conditions of the PJM, the dye concentration can be correlated to the volume fraction mixed[1]. For correct analysis, it is essential to collect and analyze several samples from the tank cavern during testing under each PJM operating condition.

The Analytical Development Section (ADS) was asked by Engineering Development Laboratory (EDL) personnel to aid in the analysis of dyed clay samples in support of this River Protection Program- Waste Treatment Program (RPP-WTP) project. These samples were originally being sent to an outside laboratory for analysis however, turn around time was insufficient and the amount of tests that were planned would generate numerous samples. ADS was asked to analyze the samples following Procedure TI-RPP-WTP-336, Dye Injection, Sampling, and Spectrometric Analysis for Non-Newtonian PJM Testing for Small Scale (4PJM) and Prototype (UFP & LS) Tanks with Sparging. Injection of the dye and sample collection was handled by EDL personnel while the analysis of the samples using an ultraviolet-visible (UV-Vis) spectrometer was performed by ADS.

More specific details of this testing, including the information on the mixing and various parameters can be found in a different report. This document only contains the actual spectrometric analysis information and the subsequent data.

2.0 Procedure

2.1 Methodology

The procedure followed for this testing, TI-RPP-WTP-336, was developed by Pacific Northwest National Laboratory (PNNL) personnel. Samples were analyzed for their absorbance and each run condition was compared for any change in the mixing. The clay was considered mixed if there was not a significant change in the absorbance reading. To determine the percent mixed volume, a series of dilutions were made for each run (from one position in the tank and the last timed sample, 5).

Each run condition (labeled A, B, C, or D along with the test number in front of the letter) had a corresponding series of dilutions, except for the final run. For each test, there were 4 different sites within the mixing tank from which samples were taken (labeled 1, 2, 3, or 4). Finally, the samples were taken every 10 minutes, for a total of 5 times (labeled 1, 2, 3, 4, or 5 with time 5 being the last sample taken). An example of the labeling is 2-4C-3, which meant this sample was from position 2 in test 4 for PJM run condition C, as the 4th sample taken. Also, an initial baseline sample was taken before a test began. This baseline was used for the dilutions and an initial reading. This baseline clay sometimes contained dye (it was clay used from the previous testing). A final sample was also taken, after the vessel was completely homogenized, usually about 30 minutes after the final time, 5.

There were many technical issues encountered at the onset of these analyses and personnel at PNNL were contacted for further instructions[2]. After correspondence, it was discovered that small details had been omitted from the procedure being followed. Examples of these issues included the amount of material needed for dilutions, sample mixing concerns including vortexing, and equilibration. After numerous conversations, the following, more detailed steps were generated for use in handling the samples along with additional important information.

1. Seven scintillation vials will be labeled with sample number, tared and recorded.
The use of plastic scintillation vials (Fisherbrand 20 mL Plastic Scintillation Vials with PP cap) makes for easier removal of all of the clay from the vial.

2. Shake sample vigorously. Using a plastic transfer pipet, place clay material from the previous run into tared and labeled vials (total volume of clay after additions should be at least 10 g). Record weight.
The dilution series must be made immediately after the samples are collected so that ideally, the 100% mixed sample and dilution series would be in contact with the baseline clay for the same amount of time. If the dilution series is not made immediately after the samples are collected, the diluted samples need to equilibrate for 48 hours before centrifuging.

3. Shake baseline clay vigorously. Calculate the appropriate amount of baseline clay to be added to the sample clay to produce dilution factors of 1.05, 1.10, 1.20, 1.40, 1.60, 1.80 and 2.00 grams. Dilution factor is calculated as the ratio of the final mass to the original sample.
With the total amount of clay in the vial being 10 grams, one can calculate the amount of initial clay needed to total 10 g for each of the dilution series. It is actually better to use a larger amount of clay to make the dilutions, however, in the beginning of testing we were limited to only a small vial of clay (<20 mL) that needed to be diluted among 8 vials. (Very early tests only used about a total of 3 grams in the dilution series. This is why there was such a problem with the linearity of the dilution curves. Ultimately the dilution series was ceased because of the linearity problem with the curves. Therefore, only a comparison of each run, not actual percent mixed, was determined, as per EDL personnel.)

4. With a plastic transfer pipet, add the appropriate amount of baseline clay to the clay in vial.
The approximate amount of baseline clay needed is calculated for a total mass of 10g. A spreadsheet was used with the measured weights to get the exact dilution factors.

5. Cap the vial, shake vigorously to mix and let sit for at least 24 hours.
The clay needed to sit for 24 hours to allow for equilibration.

6. Shake diluted sample vial. Transfer the diluted clay with a transfer pipet to centrifuge vial. Fill about 1/3 to ½ full. Cap the vial. Also transfer other samples not used in dilution series.
(Kimble Glass 10mL Conical Bottom Glass Centrifuge Tubes with phenolic cap)

7. Vortex the dilution series samples for 3 minutes to ensure a thorough mix.
The vortexing can either be performed while the sample is in the scintillation vial, or after it is transferred to the centrifuge vial.

8. Centrifuge the sample for 50-60 minutes. If the liquid is not clear of particulates, centrifuge longer.
When looking at the supernate liquid, one can identify a clear solution versus one with particulates floating around which may interfere with the UV-Vis absorption reading.

9. Transfer supernate liquid on top of compacted solid with a diposable glass pipet to a plastic cuvette.
(Fisherbrand Semi UV 1.5 mL Cuvette with cap)

10. Zero instrument using deionized water. Place cuvette in UV-Vis sample holder.

11. Analyze absorbance at ~630 nm. Record absorbance at 630 nm and at a wavelength outside the absorbance range of the dye (~730nm). Subtract the two for the absorbance reading.

12. If the absorbance is greater than 1.5, dilute the supernate with deionized water. Record the amount of supernate and water used in the dilution.

The spectrometer (J&M Tidas Spectrometer with Ocean Optics Inc. CUV Sample Holder) software (Tidas Spectralys 2.0 Software) was set to average 30 spectra with a 10 ms exposure. The light source (Ocean Optics Inc. LS-1 tungsten halogen light source) was monitored every 8 samples for drift.

Appropriate quality assurance controls were taken to ensure proper measurements. The balance (Denver Instruments M-310 Balance, MT&E no. AD-0075) was checked and calibrated when needed, and recorded in the MT&E #AD-0075 control log. The spectrometer was referenced with deionized water, after the instrument had equilibrated for 30 minutes, every 8th sample and after every test and a new dark current spectra was taken at this time too. The instrument was monitored for any drift in the light source or fiber alignment issues during those times also.

2.2 Data Analysis

The absorbance versus dilution factor is plotted for set A of the dilution samples. On this plot, the undiluted sample from set A is also plotted as dilution factor 1.0 (it should be the highest absorbance reading). Also on this plot, from its absorbance reading, place the original sample from set B. From this point, interpolate the dilution factor in the PJM vessel. Next, plot the dilution series from set B along with the undiluted set B sample. On this plot, place the absorbance for the undiluted set C sample and interpolate the dilution factor. Do this for set C and set D. The final homogenized sample absorbance reading will be plotted on the curve from set D and the dilution factor will be interpolated. This method minimizes problems associated with the mass balance dye method by re-baselining the dye concentrations at each run condition. To calculate volume mixed in each run, remember that the dilution factor is equal to the final mass divided by the initial sample mass. For Run D, the final homogenized sample's interpolated dilution factor is equal to 100% divided by the unknown initial sample mass. This will be the percent mixed for run D. Next, the undiluted sample from run D that was plotted on the run C curve is the dilution factor and that is equal to the percentage calculated previously (percent mixed for run D) divided by the unknown initial sample mass. This percent mixed will be used to calculate for the unknown initial sample mass for the B run and then this percent mixed will be used to calculate the mixed percent for run A. An example of this can be found in Appendix A.

For all other samples collected, a simple bar graph indicates the change of mixing for the various times and sampling points. Standard deviation was calculated for some samples and is indicated by error bars on the graphs. (In the beginning of testing, multiple samples were not run for each test because there were so many samples, results were needed and the focus was on understanding the technical issues with the dilution sets. As the tests continued however, multiple samples were taken from a vial to calculate for standard deviation, 3σ.)

3.0 RESULTS

On each of the graphs, the absorbance versus time, is plotted for each run condition for a particular position (1, 2, 3, or 4). The bar graphs begin with the starting clay (the initial baseline absorbance, I), show the absorbance over the various times collected (1-5) and end with the final absorbance (F) of the final homogenized clay for that particular test. The results given here can be found in WSRC-NB-2004-00012[3]. The dilution curves are not contained in this report, but can be found in the Notebook as well.

3.1 Test 1, 10 Pa Figure 1, Table 1 1/14/04

Test 1 only contains information for the first time collected and the fifth time collected.

3.2 Test 2, 20 Pa Figure 2, Table 2 1/15/04

Test 2 contains information collected over the entire test period. The very low initial (I) absorbance indicates the use of undyed clay when staring the test.

3.3 Test 3, 10 Pa Figure 3, Table 3 1/27/04

Test 3 contains only information for the first and fifth times collected.

3.4 Test 4, 20 Pa Figure 4, Table 4 1/28/04

Test 4 contains only information for the first and fifth times collected.

3.5 Test 5, 30 Pa Figure 5, Table 5 1/29/04

Test 5 contains information for the entire time collected for the first position and only the first and fifth times collected for the remaining positions. The initial absorbance (I) reading indicates the use of fresh undyed clay.

3.6 Test 6, 30 Pa Figure 6, Table 6 1/31/04

Test 6 data only contains the information for the fifth reading. Problems were still being encountered with the dilution curves and with the samples being thoroughly mixed. These issues were being resolved and only the last collection and final mixed clay absorbances were requested.

3.7 Test 7, 30 Pa Figure 7, Table 7 2/6/04

Test 7 data contains the first, third and fifth collection times. Again, undyed clay was used at the onset of this test.

3.8 Test 8, 30 Pa Figure 8, Table 8 2/9/04

Test 8 data contains information collected over the entire test period. For this test however, only three different conditions were used (A, B, and C).

3.9 Test 9, Figure 9, Table 9 2/10/04

Test 9 data contains information collected over the entire test period. For this test only three different conditions were used (A, B, and C).

3.10 Test 10, Figure 10, Table 10 2/12/04

Test 10 data contains information collected over the entire test period. For this test only three different conditions were used (A, B, and C).

3.11 Test 11, Figure 11, Table 11 2/13/04

Test 11 data contains information collected over the entire test period. For this test only three different conditions were used (A, B, and C).

3.12 Test 12, Figure 12, Table 12 2/24/04

Test 12 contains data collected for only three times and following one condition (A) only. Times 2, 3 and final are statistically the same absorbance reading.

3.13 Test 13, Figure 13, Table 13 2/25/04

Test 13 contains data collected for only three times and following one condition (A) only. Times 2, 3 and final are statistically the same absorbance reading.

3.14 Test 14, Figure 14, Table 14 3/15/04

Test 14 contains data collected for only three times and following one condition (A) only. Times 2, 3 and final are statistically the same absorbance reading.

3.15 Test 15, Figure 15, Table 15 3/22/04

Test 15 contains data collected for only three times and following one condition (A) only. Times 2, 3 and final are statistically the same absorbance reading.

4.0 CONCLUSIONS

This document contains all the absorption data requested for the pulse jet mixing tests and has been used in data analysis by EDL personnel. Many technical issues were encountered with the original procedure that was given for this work. After many discussions with EDL and PNNL personnel, a more detailed procedure was produced. However, the dilution curves were still a problem with results varying and the typical trend not being followed.

One of the technical issues discussed was the introduction of dye to the tank. ADS personnel believed that one sample homogenization was a major concern. Upon opening the sample vials, swirls of blue dye could be viewed which was an indication that the dye was not mixed properly before being distributed to the tank. We believe if the dye was introduced as a solution, that is completely dissolved into water before introduction, the blending of the tank would have been more effiecient. EDL personnel indicated that due to mass balance issues, nothing more could be introduced into the tank. One possible solution discussed was to centrifuge the initial tank clay and siphon off the liquid to mix with the dye. This however did not work as planned because not enough liquid could by siphoned off to mix with the large amount of dye. After this attempt, it was decided to take a certain amount of the initial clay and mix it in a blender with the dye. This did not completely solve the mixing issue but did help.

This report contains the information given to EDL personnel for their data interpretation. Although the intention of this work was to determine the percentage mixed within a tank under various conditions, because of the technical issues encountered with the original procedure, it does not report the percent mixed, but only addresses absorption data for all the samples. From this, an improved, more detailed procedure was developed through discussions with PNNL and EDL personnel. Useful data from the sample absorption graphs were obtained for analysis.

5.0 ACKNOWLEDGEMENTS

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6.0 REFERENCES

1. Procedure TI-RPP-WTP-336, Dye Injection, Sampling, and Spectrometric Analysis for Non-Newtonian PJM Testing for Small Scale (4PJM) and Prototype (UFP & LS) Tanks with Sparging
2. Personal correspondence with Adam Poloski, PNNL Scientist, 1/15/04, 1/28/04, 2/2/04, 2/6/04, 2/12/04.
3. Zeigler, K. Clay Analysis, WSRC-NB-2004-00012.

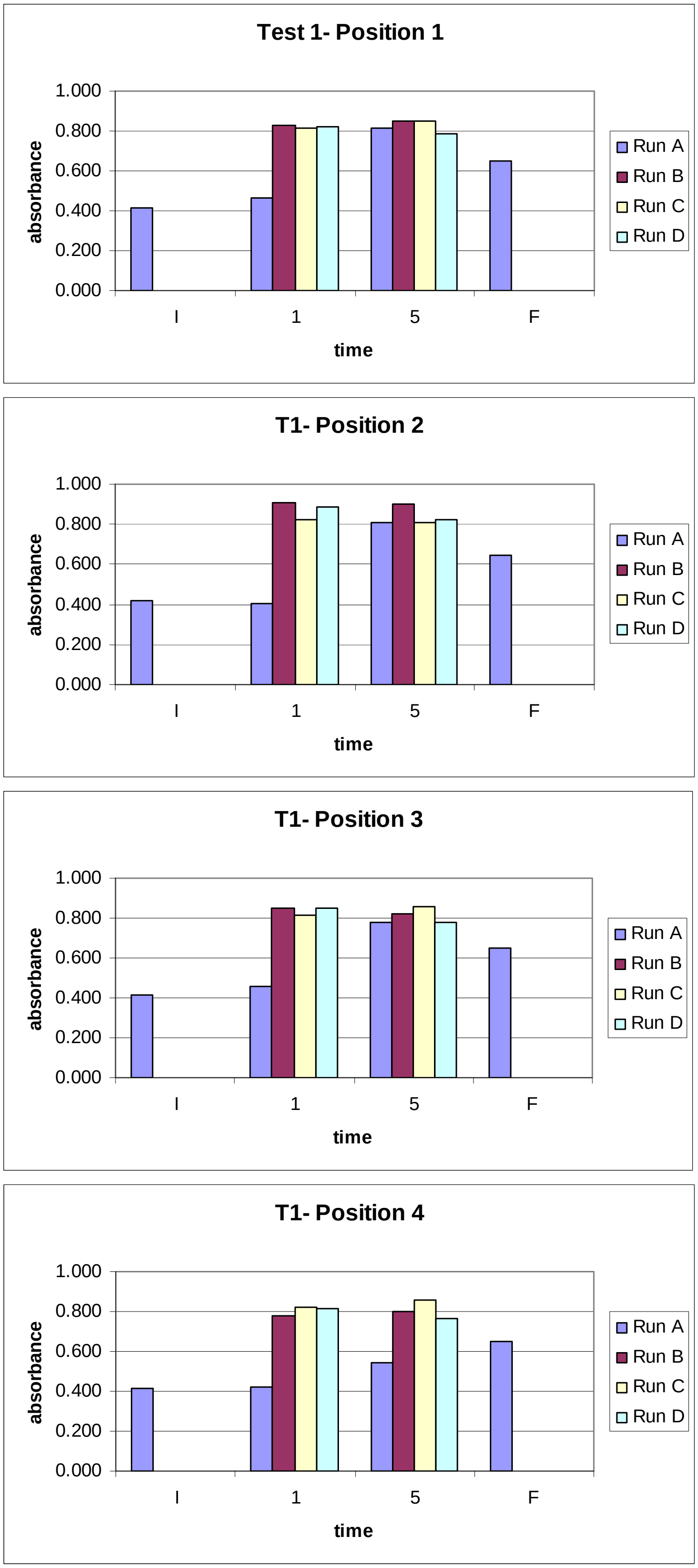


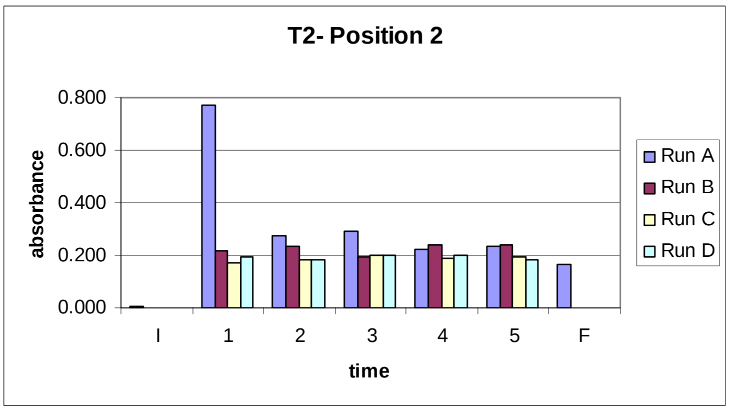
Figure 1. Test 1

Test 1

Sample	Abs	**Sample**	Abs	**Sample**	Abs	**Sample**	Abs
DI	0.415						
1A1	0.461	1B1	0.831	1C1	0.812	1D1	0.820
1A5	0.812	1B5	0.849	1C5	0.853	1D5	0.787
DF	0.648						
DI	0.415						
2A1	0.405	2B1	0.905	2C1	0.820	2D1	0.890
2A5	0.809	2B5	0.904	2C5	0.812	2D5	0.822
DF	0.648						
DI	0.415						
3A1	0.459	3B1	0.848	3C1	0.817	3D1	0.851
3A5	0.777	3B5	0.821	3C5	0.856	3D5	0.776
DF	0.648						
DI	0.415						
4A1	0.419	4B1	0.781	4C1	0.824	4D1	0.816
4A5	0.546	4B5	0.801	4C5	0.859	4D5	0.765
DF	0.648						

Table 1. Test 1


Test 2- Position 1
absorbance
0.800
0.600
0.400
0.200
0.000
I
1
2
3
4
5
F
time
Run A
Run B
Run C
Run D


T2- Position 2
absorbance
0.800
0.600
0.400
0.200
0.000
I
1
2
3
4
5
F
time
Run A
Run B
Run C
Run D


T2- Position 3
absorbance
0.800
0.600
0.400
0.200
0.000
I
1
2
3
4
5
F
time
Run A
Run B
Run C
Run D

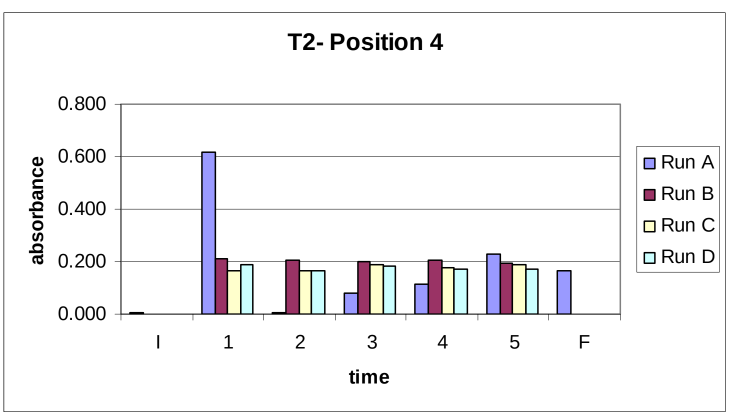

T2- Position 4
absorbance
0.800
0.600
0.400
0.200
0.000
I
1
2
3
4
5
F
time
Run A
Run B
Run C
Run D

Figure 2. Test 2

Test 2-20Pa

Sample	Abs	Samp	Abs	Samp	Abs	Samp	Abs
DI	0.005						
12A1	0.697	12B1	0.209	12C1	0.231	12D1	0.208
12A2	0.211	12B2	0.217	12C2	0.206	12D2	0.223
12A3	0.193	12B3	0.220			12D3	0.202
12A4	0.193	12B4	0.215	12C4	0.203	12D4	0.168
12A5	0.187	12B5	0.204	12C5	0.208	12D5	0.199
DF	0.167						
DI	0.005						
22A1	0.770	22B1	0.216	22C1	0.172	22D1	0.195
22A2	0.276	22B2	0.236	22C2	0.182	22D2	0.180
22A3	0.290	22B3	0.196	22C3	0.201	22D3	0.201
22A4	0.225	22B4	0.242	22C4	0.187	22D4	0.202
22A5	0.236	22B5	0.242	22C5	0.192	22D5	0.182
DF	0.167						
DI	0.005						
32A1	0.748	32B1	0.217	32C1	0.186	32D1	0.188
32A2	0.317	32B2	0.208	32C2	0.225	32D2	0.177
32A3	0.242	32B3	0.207	32C3	0.194	32D3	0.199
32A4	0.225	32B4	0.088	32C4	0.193	32D4	0.191
32A5	0.227	32B5	0.197	32C5	0.181	32D5	0.190
DF	0.167						
DI	0.005						
42A1	0.616	42B1	0.212	42C1	0.168	42D1	0.190
42A2	0.004	42B2	0.203	42C2	0.165	42D2	0.167
42A3	0.080	42B3	0.202	42C3	0.186	42D3	0.182
42A4	0.112	42B4	0.204	42C4	0.178	42D4	0.169
42A5	0.226	42B5	0.193	42C5	0.187	42D5	0.169
DF	0.167						

Table 2. Test 2

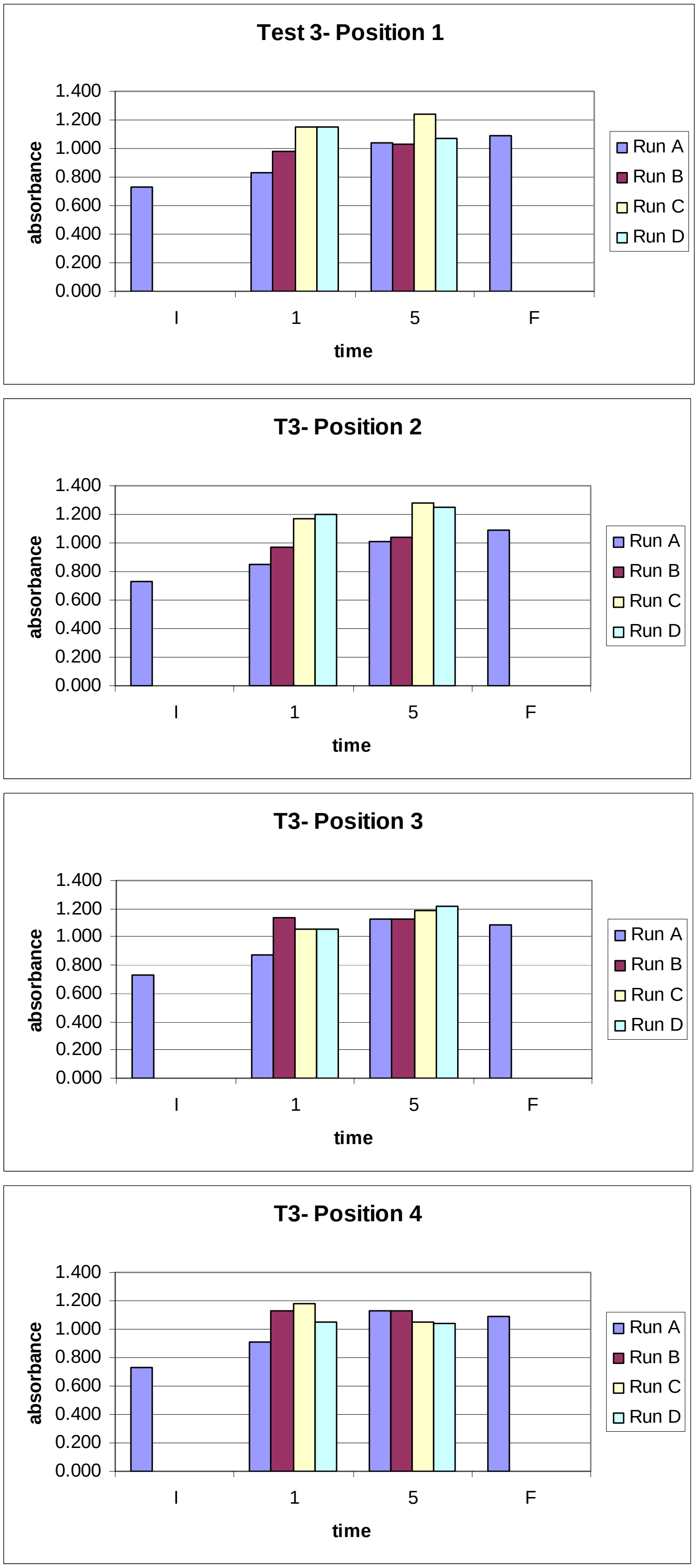
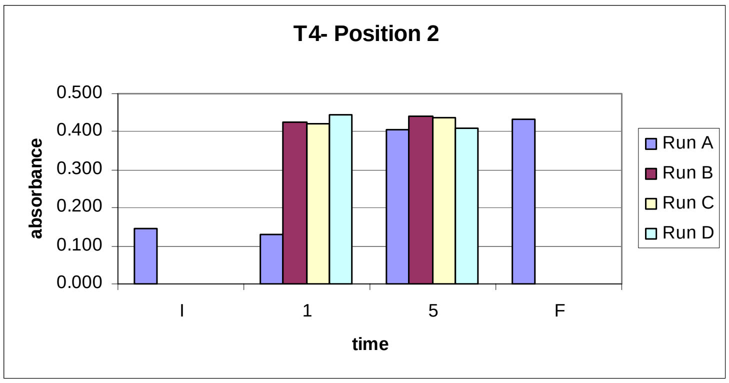


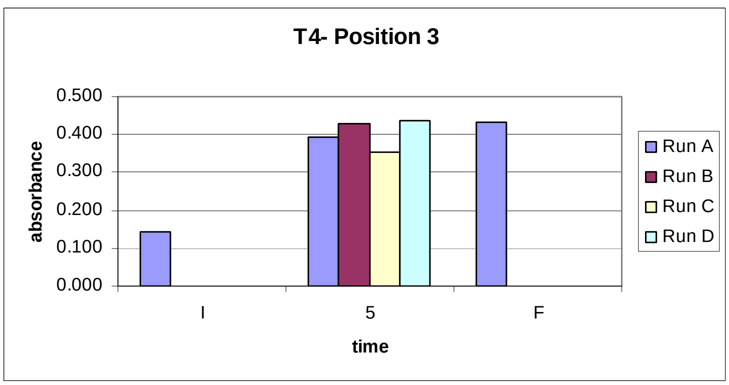
Figure 3. Test 3

Test 3

Sample	Abs	Samp	Abs	Samp	Abs	Samp	Abs
DI	0.730						
1A1	0.831	1B1	0.983	1C1	1.146	1D1	1.153
1A5	1.042	1B5	1.035	1C5	1.235	1D5	1.072
DF	1.089						
DI	0.730						
2A1	0.854	2B1	0.974	2C1	1.166	2D1	1.198
2A5	1.012	2B5	1.042	2C5	1.276	2D5	1.255
DF	1.089						
DI	0.730						
3A1	0.876	3B1	1.132	3C1	1.053	3D1	1.056
3A5	1.122	3B5	1.129	3C5	1.185	3D5	1.213
DF	1.089						
DI	0.730						
4A1	0.914	4B1	1.131	4C1	1.176	4D1	1.053
4A5	1.131	4B5	1.130	4C5	1.055	4D5	1.043
DF	1.089						

Table 3. Test 3





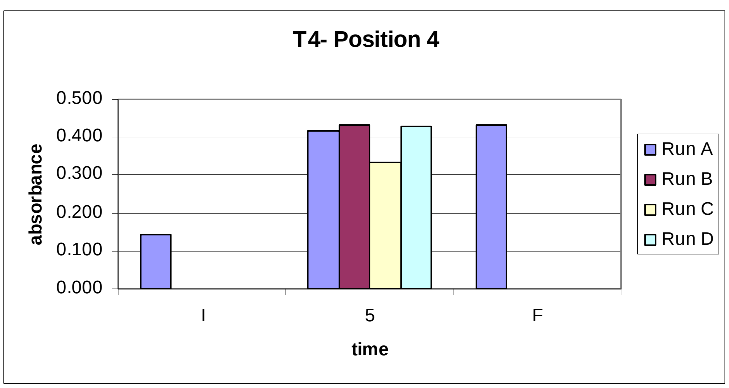


Figure 4. Test 4

Test 4							
Sample	**Abs**	**Sample**	**Abs**	**Sample**	**Abs**	**Sample**	**Abs**
DI	0.145						
12A5	0.391	12B5	0.424	12C5	0.422	12D5	0.426
DF	0.434						
DI	0.145						
22A1	0.13	22B1	0.427	22C1	0.421	22D1	0.445
22A5	0.405	22B5	0.440	22C5	0.435	22D5	0.410
DF	0.434						
DI	0.145						
32A5	0.392	32B5	0.430	32C5	0.353	32D5	0.436
DF	0.434						
DI	0.145						
42A5	0.417	42B5	0.431	42C5	0.334	42D5	0.428
DF	0.434						

Table 4. Test 4

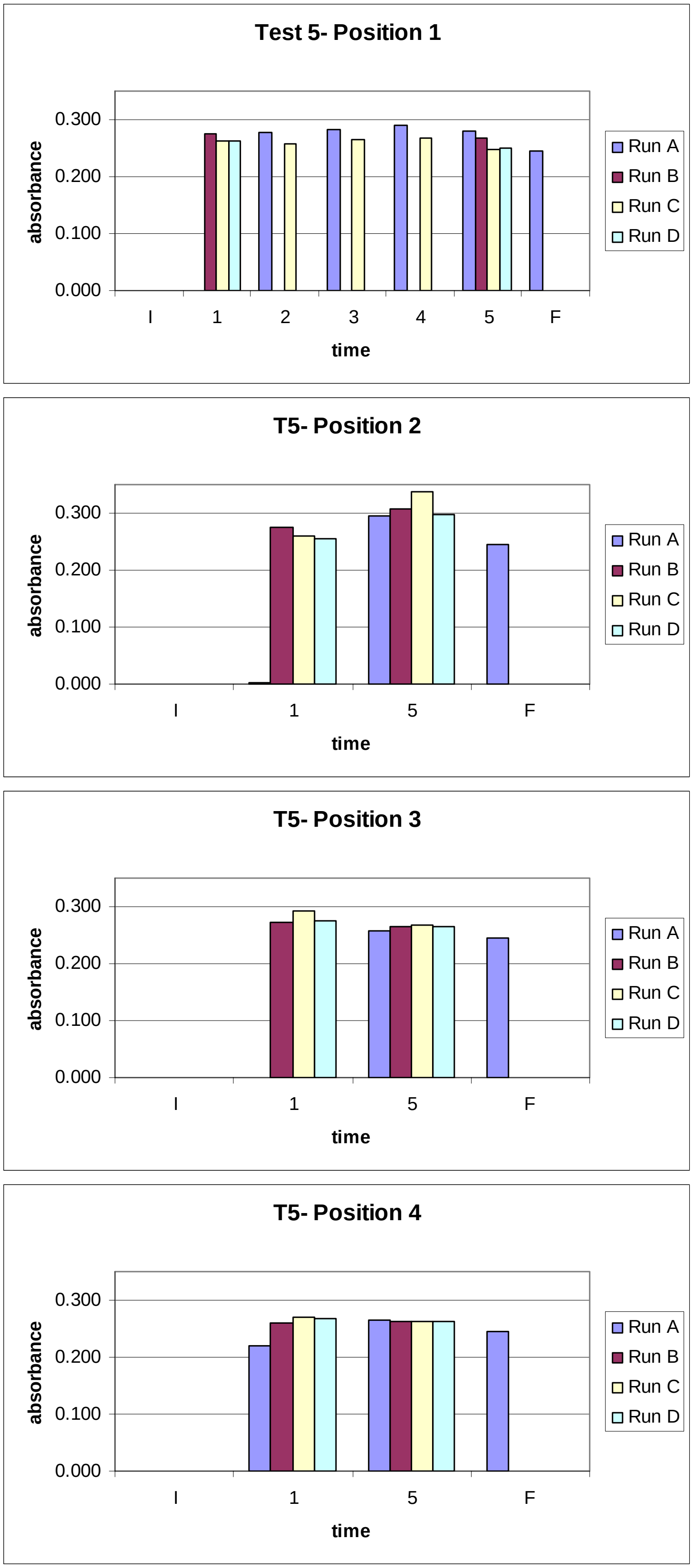


Figure 5. Test 5

Test 5- 30 Pa

Sample	Abs	Sample	Abs	Sample	Abs	Sample	Abs
DI	0.001						
13A1	0.001	13B1	0.274	13C1	0.262	1D1	0.263
13A2	0.279			13C2	0.256		
13A3	0.282			13C3	0.265		
13A4	0.291			13C4	0.268		
13A5	0.281	13B5	0.268	13C5	0.249	1D5	0.250
Fin	0.244						
DI	0.001						
23A1	0.001	23B1	0.274	2C1	0.259	2D1	0.256
23A5	0.294	23B5	0.306	2C5	0.337	2D5	0.297
DF	0.244						
DI	0.001						
33A1	0.001	33B1	0.272	3C1	0.292	3D1	0.274
33A5	0.257	33B5	0.265	3C5	0.267	3D5	0.265
DF	0.244						
DI	0.001						
43A1	0.221	43B1	0.259	4C1	0.269	4D1	0.266
43A5	0.265	43B5	0.263	4C5	0.262	4D5	0.264
DF	0.244						

Table 5. Test 5

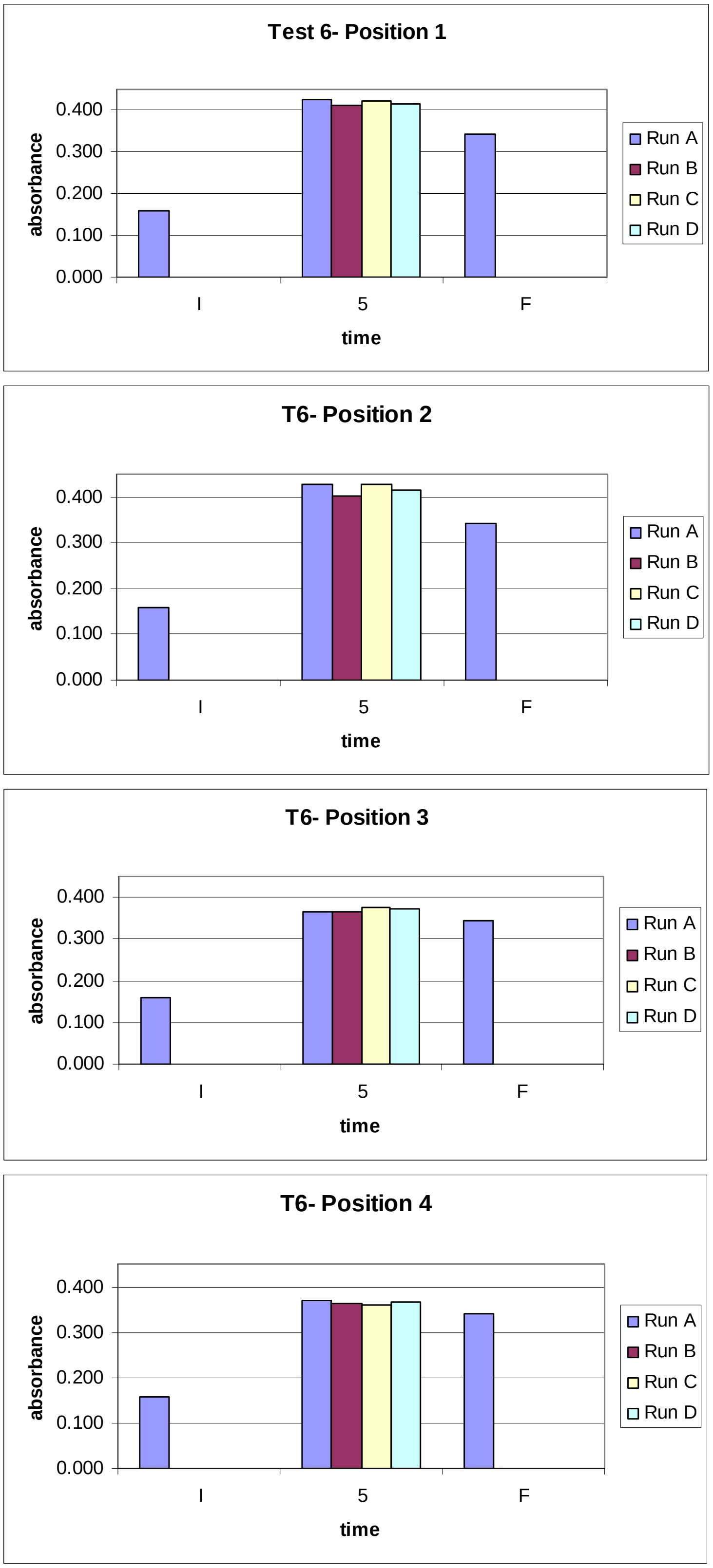


Figure 6. Test 6

Test 6 Sample	Abs	Sample	Abs	Sample	Abs	Sample	Abs
DI	0.158						
14a5	0.425	14b5	0.413	14C5	0.423	14D5	0.414
DF	0.342						
DI	0.158						
24a5	0.427	24b5	0.403	24C5	0.427	24D5	0.415
DF	0.342						
DI	0.158						
34a5	0.365	34b5	0.365	34C5	0.377	34D5	0.372
DF	0.342						
DI	0.158						
44a5	0.369	44b5	0.363	44C5	0.361	44D5	0.367
DF	0.342						

Table 6. Test 6

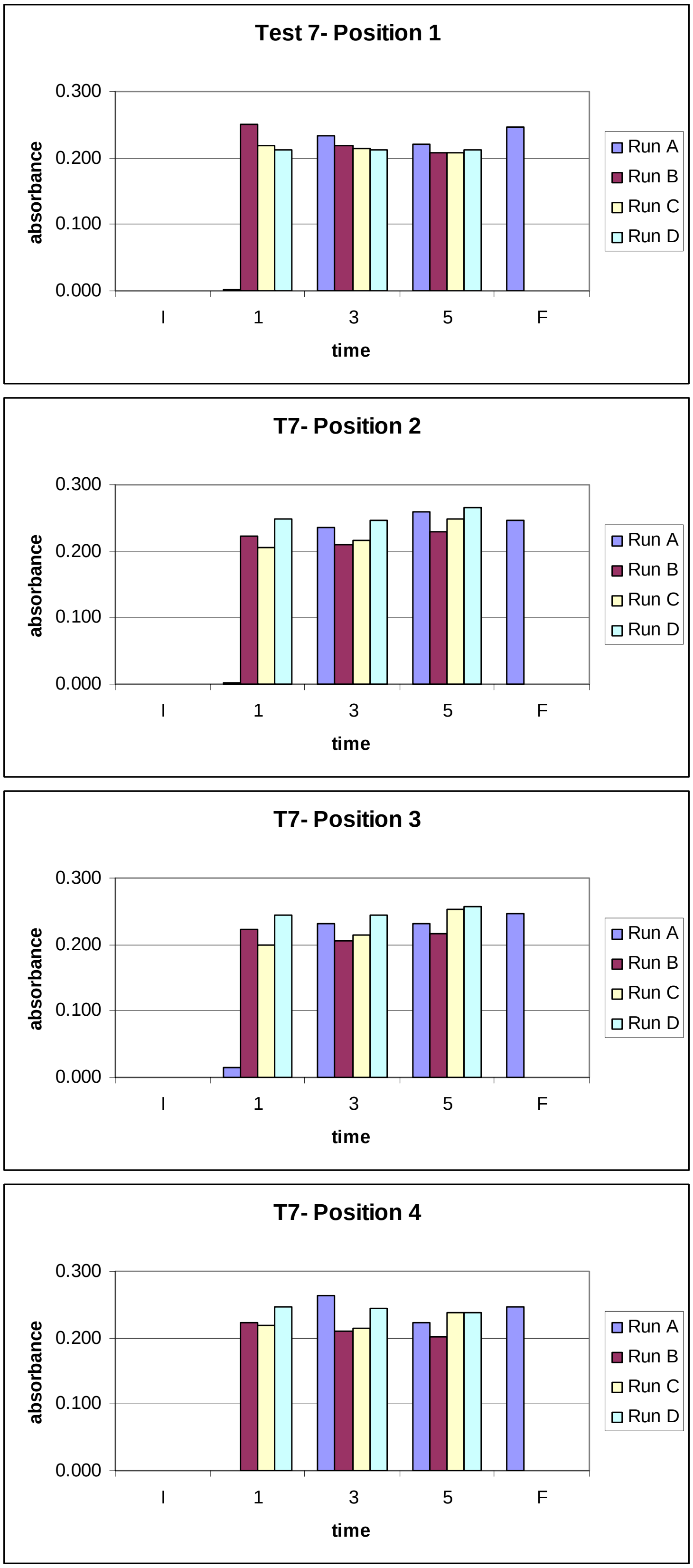


Figure 7. Test 7

Test 7 Sample	Abs	Samp	Abs	Samp	Abs	Samp	Abs
DI	0.001						
17A1	0.002	17B1	0.250	17C1	0.218	17D1	0.211
17A3	0.234	17B3	0.219	17C3	0.213	17D3	0.212
17A5	0.221	17B5	0.208	17C5	0.207	17D5	0.212
DF	0.245						
DI	0.001						
27A1	0.003	27B1	0.223	27C1	0.205	27D1	0.249
27A3	0.235	27B3	0.210	27C3	0.217	27D3	0.246
27A5	0.260	27B5	0.229	27C5	0.249	27D5	0.266
DF	0.245						
DI	0.001						
37A1	0.015	37B1	0.224	37C1	0.200	37D1	0.243
37A3	0.231	37B3	0.206	37C3	0.214	37D3	0.244
37A5	0.232	37B5	0.216	37C5	0.254	37D5	0.256
DF	0.245						
DI	0.001						
47A1	0.001	47B1	0.222	47C1	0.219	47D1	0.247
47A3	0.263	47B3	0.210	47C3	0.214	47D3	0.244
47A5	0.222	47B5	0.201	47C5	0.239	47D5	0.238
DF	0.245						

Table 7. Test 7

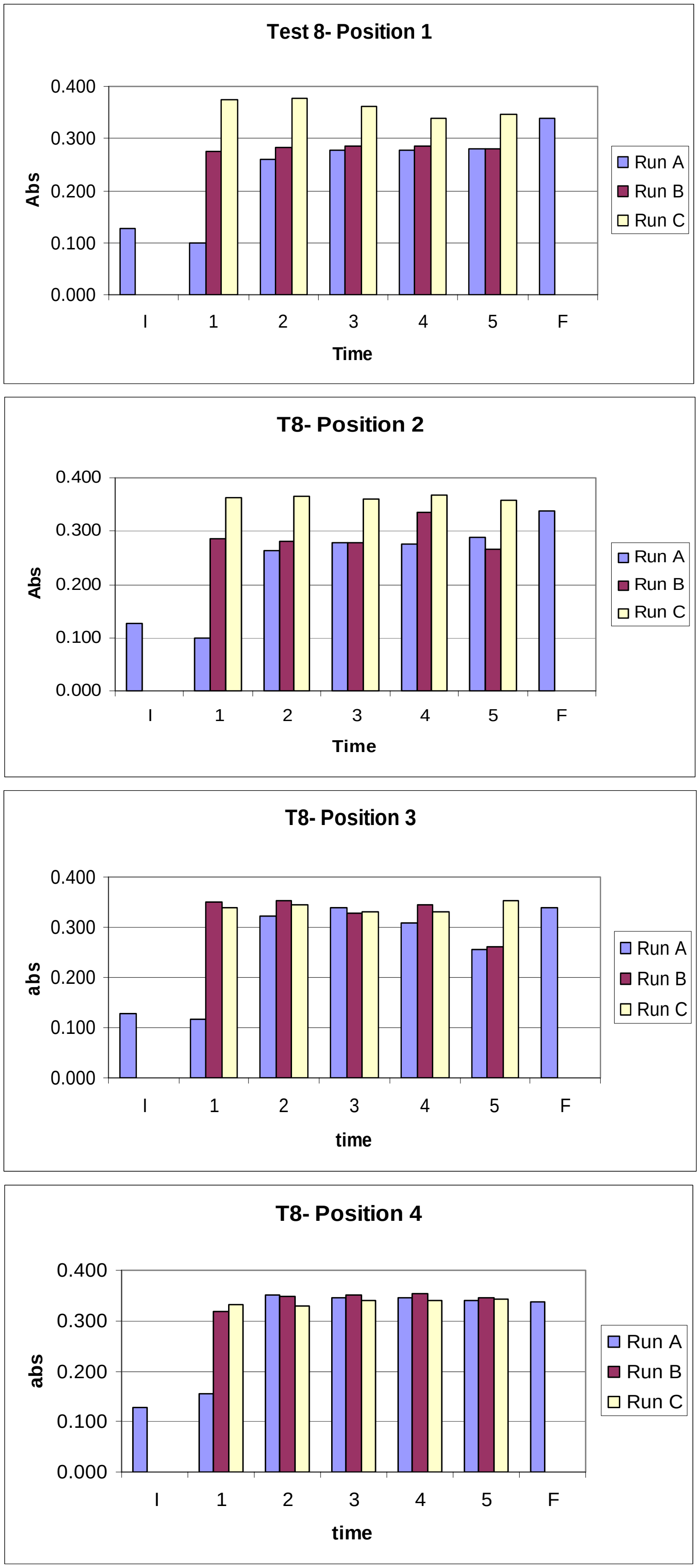


Figure 8. Test 8

Test 8 Sample	Abs	Sample	Abs	Sample	Abs
I	0.127				
18A1	0.099	18B1	0.274	18C1	0.374
18A2	0.259	18B2	0.283	18C2	0.376
18A3	0.279	18B3	0.285	18C3	0.362
18A4	0.278	18B4	0.286	18C4	0.339
18A5	0.282	18B5	0.280	18C5	0.347
FIN	0.338				
I	0.127				
28A1	0.100	28B1	0.285	28C1	0.362
28A2	0.264	28B2	0.280	28C2	0.366
28A3	0.278	28B3	0.277	28C3	0.360
28A4	0.277	28B4	0.334	28C4	0.367
28A5	0.287	28B5	0.266	28C5	0.359
FIN	0.338				
I	0.127				
38A1	0.116	38B1	0.351	38C1	0.339
38A2	0.323	38B2	0.353	38C2	0.344
38A3	0.338	38B3	0.328	38C3	0.330
38A4	0.309	38B4	0.345	38C4	0.331
38A5	0.256	38B5	0.262	38C5	0.353
FIN	0.338				
I	0.127				
48A1	0.156	48B1	0.319	48C1	0.331
48A2	0.350	48B2	0.348	48C2	0.330
48A3	0.346	48B3	0.352	48C3	0.339
48A4	0.345	48B4	0.353	48C4	0.341
48A5	0.340	48B5	0.346	48C5	0.342
FIN	0.338				

Table 8. Test 8

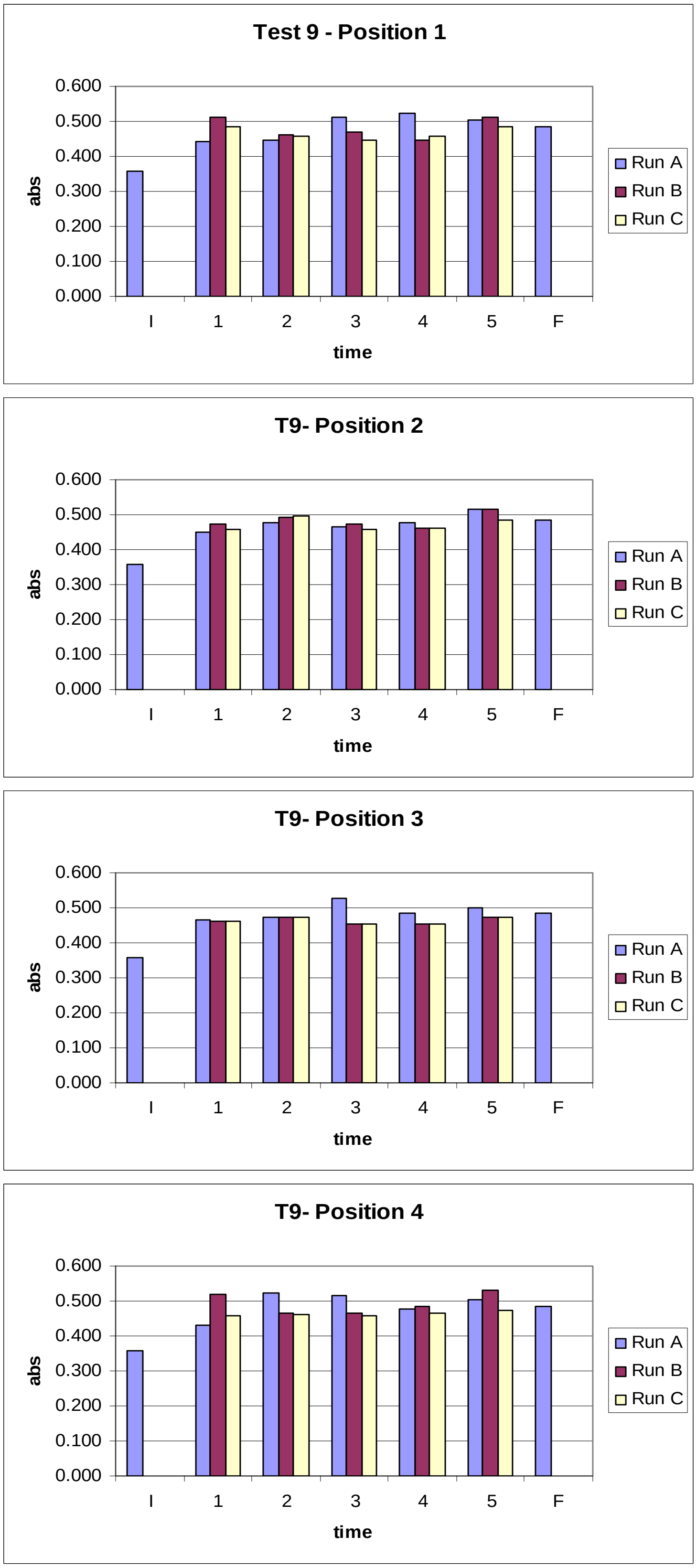


Figure 9. Test 9

Test 9					
Sample	**Abs**	**Sample**	**Abs**	**Sample**	**Abs**
Ini	0.359				
19A1	0.444	19B1	0.512	19C1	0.485
19A2	0.445	19B2	0.461	19C2	0.458
19A3	0.511	19B3	0.468	19C3	0.446
19A4	0.525	19B4	0.446	19C4	0.459
19A5	0.505	19B5	0.510	19C5	0.486
Fin	0.485				
Ini	0.359				
29A1	0.452	29B1	0.471	29C1	0.456
29A2	0.477	29B2	0.493	29C2	0.498
29A3	0.467	29B3	0.472	29C3	0.458
29A4	0.475	29B4	0.462	29C4	0.461
29A5	0.515	29B5	0.516	29C5	0.485
Fin	0.485				
Ini	0.359				
39A1	0.464	39B1	0.496	39C1	0.462
39A2	0.472	39B2	0.469	39C2	0.475
39A3	0.526	39B3	0.471	39C3	0.455
39A4	0.485	39B4	0.532	39C4	0.454
39A5	0.499	39B5	0.513	39C5	0.474
Fin	0.485				
Ini	0.359				
49A1	0.430	49B1	0.519	49C1	0.456
49A2	0.524	49B2	0.465	49C2	0.462
49A3	0.514	49B3	0.465	49C3	0.457
49A4	0.477	49B4	0.486	49C4	0.467
49A5	0.504	49B5	0.532	49C5	0.473
Fin	0.485				

Table 9. Test 9

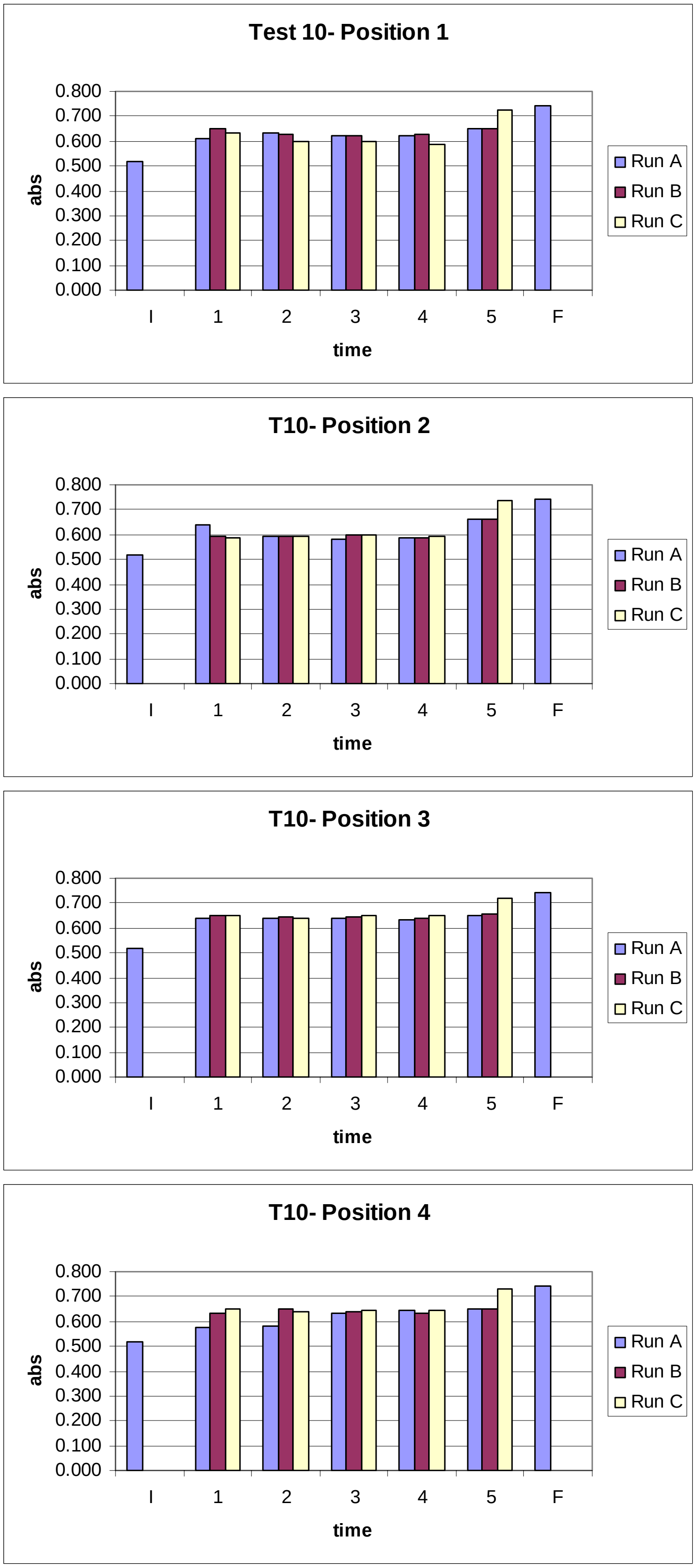


Figure 10. Test 10

Test 10 Sample	Abs	Sample	Abs	Sample	Abs
Initial	0.520				
110A1	0.608	110B1	0.650	110C1	0.634
110A2	0.636	110B2	0.630	110C2	0.596
110A3	0.619	110B3	0.621	110C3	0.596
110A4	0.621	110B4	0.625	110C4	0.586
110A5	0.652	110B5	0.649	110C5	0.723
final	0.740				
Initial	0.520				
210A1	0.638	210B1	0.591	210C1	0.589
210A2	0.593	210B2	0.595	210C2	0.592
210A3	0.584	210B3	0.596	210C3	0.601
210A4	0.585	210B4	0.589	210C4	0.592
210A5	0.661	210B5	0.659	210C5	0.738
final	0.740				
Initial	0.520				
310A1	0.639	310B1	0.649	310C1	0.650
310A2	0.636	310B2	0.646	310C2	0.641
310A3	0.639	310B3	0.642	310C3	0.648
310A4	0.636	310B4	0.641	310C4	0.648
310A5	0.652	310B5	0.653	310C5	0.721
final	0.740				
Initial	0.520				
410A1	0.573	410B1	0.635	410C1	0.649
410A2	0.580	410B2	0.651	410C2	0.637
410A3	0.634	410B3	0.636	410C3	0.647
410A4	0.642	410B4	0.635	410C4	0.646
410A5	0.648	410B5	0.651	410C5	0.730
final	0.740				

Table 10. Test 10

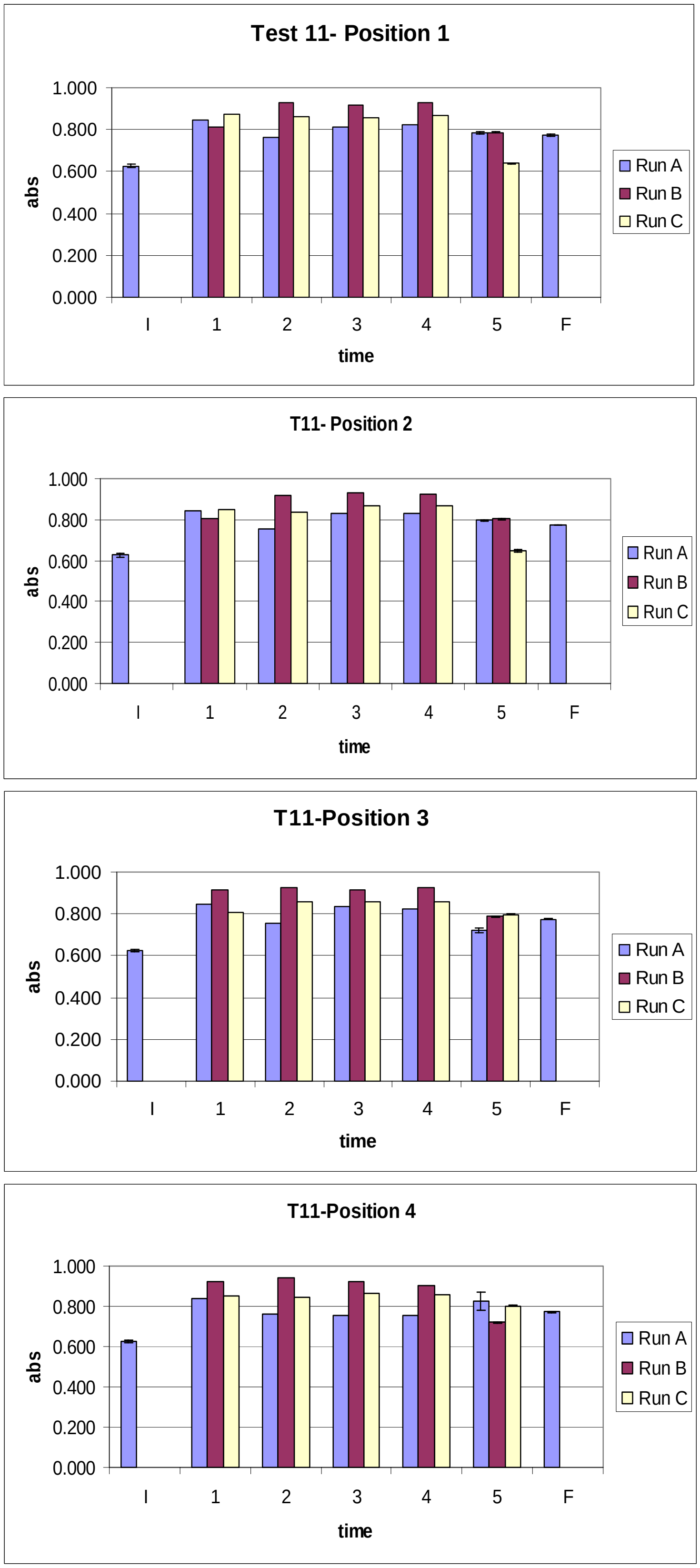
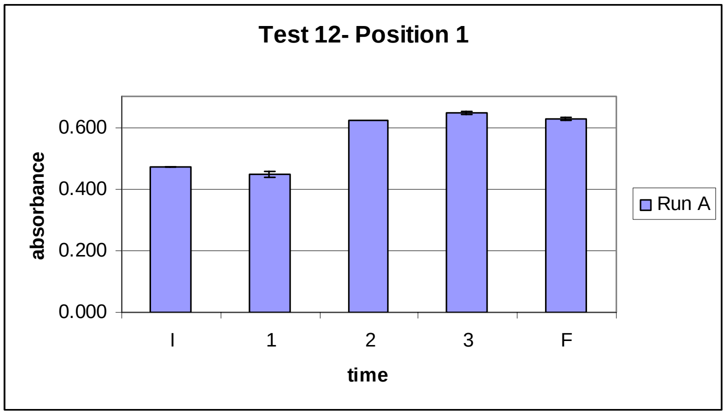
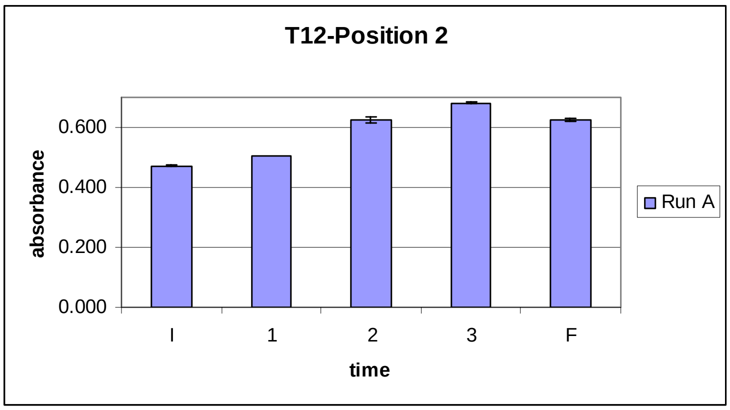


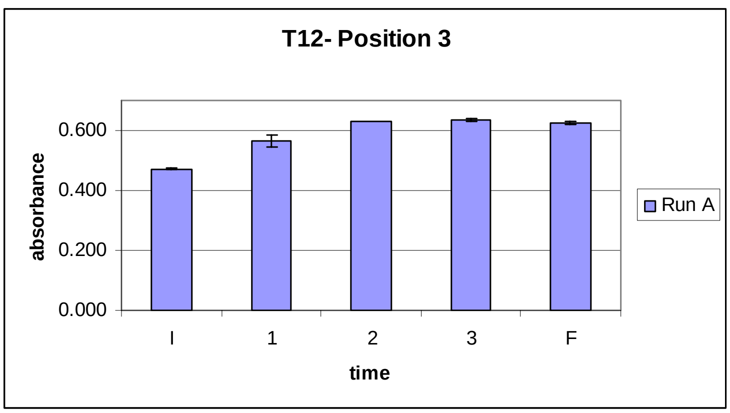
Figure 11. Test 11

Test 11 Samp	Abs	dev	Samp	Abs	dev	Samp	Abs	dev	Samp	Abs	dev
Ini	0.626	0.008	Ini	0.626	0.008	Ini	0.626	0.008	Ini	0.626	0.008
111A1	0.847		211A1	0.842		311A1	0.846		411A1	0.841	
111A2	0.762		211A2	0.757		311A2	0.757		411A2	0.759	
111A3	0.813		211A3	0.832		311A3	0.833		411A3	0.758	
111A4	0.824		211A4	0.831		311A4	0.823		411A4	0.758	
111A5	0.785	0.005	211A5	0.796	0.001	311A5	0.723	0.011	411A5	0.826	0.043
Fin	0.773	0.003	Fin	0.773	0.003	Fin	0.773	0.003	Fin	0.773	0.003
111B1	0.814		211B1	0.804		311B1	0.913		411B1	0.921	
111B2	0.926		211B2	0.917		311B2	0.926		411B2	0.942	
111B3	0.915		211B3	0.928		311B3	0.914		411B3	0.920	
111B4	0.929		211B4	0.925		311B4	0.926		411B4	0.906	
111B5	0.786	0.002	211B5	0.802	0.001	311B5	0.788	0.003	411B5	0.720	0.001
111C1	0.871		211C1	0.850		311C1	0.808		411C1	0.851	
111C2	0.862		211C2	0.839		311C2	0.856		411C2	0.846	
111C3	0.856		211C3	0.867		311C3	0.861		411C3	0.863	
111C4	0.867		211C4	0.869		311C4	0.858		411C4	0.856	
111C5	0.639	0.001	211C5	0.648	0.006	311C5	0.797	0.003	411C5	0.803	0.005

Table 11. Test 11


Test 12- Position 1
absorbance
0.600
0.400
0.200
0.000
I
1
2
3
F
time
Run A


T12-Position 2
absorbance
0.600
0.400
0.200
0.000
I
1
2
3
F
time
Run A


T12- Position 3
absorbance
0.600
0.400
0.200
0.000
I
1
2
3
F
time
Run A

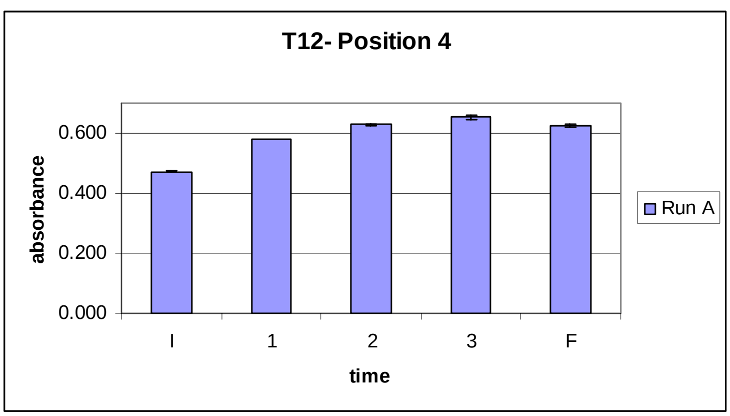

T12- Position 4
absorbance
0.600
0.400
0.200
0.000
I
1
2
3
F
time
Run A

Figure 12. Test 12

Test 12

Sample	Abs	dev
I	0.471	0.001
1-12-A-1	0.447	0.008
1-12-A-2	0.620	
1-12-A-3	0.646	0.003
Fin	0.625	0.005
I	0.471	0.001
2-12-A-1	0.507	
2-12-A-2	0.625	0.008
2-12-A-3	0.681	0.002
Fin	0.625	0.005
I	0.471	0.001
3-12-A-1	0.56	0.02
3-12-A-2	0.630	
3-12-A-3	0.636	0.004
Fin	0.625	0.005
I	0.471	0.001
4-12-A-1	0.580	
4-12-A-2	0.629	0.003
4-12-A-3	0.653	0.008
Fin	0.625	0.005

Table 12. Test 12

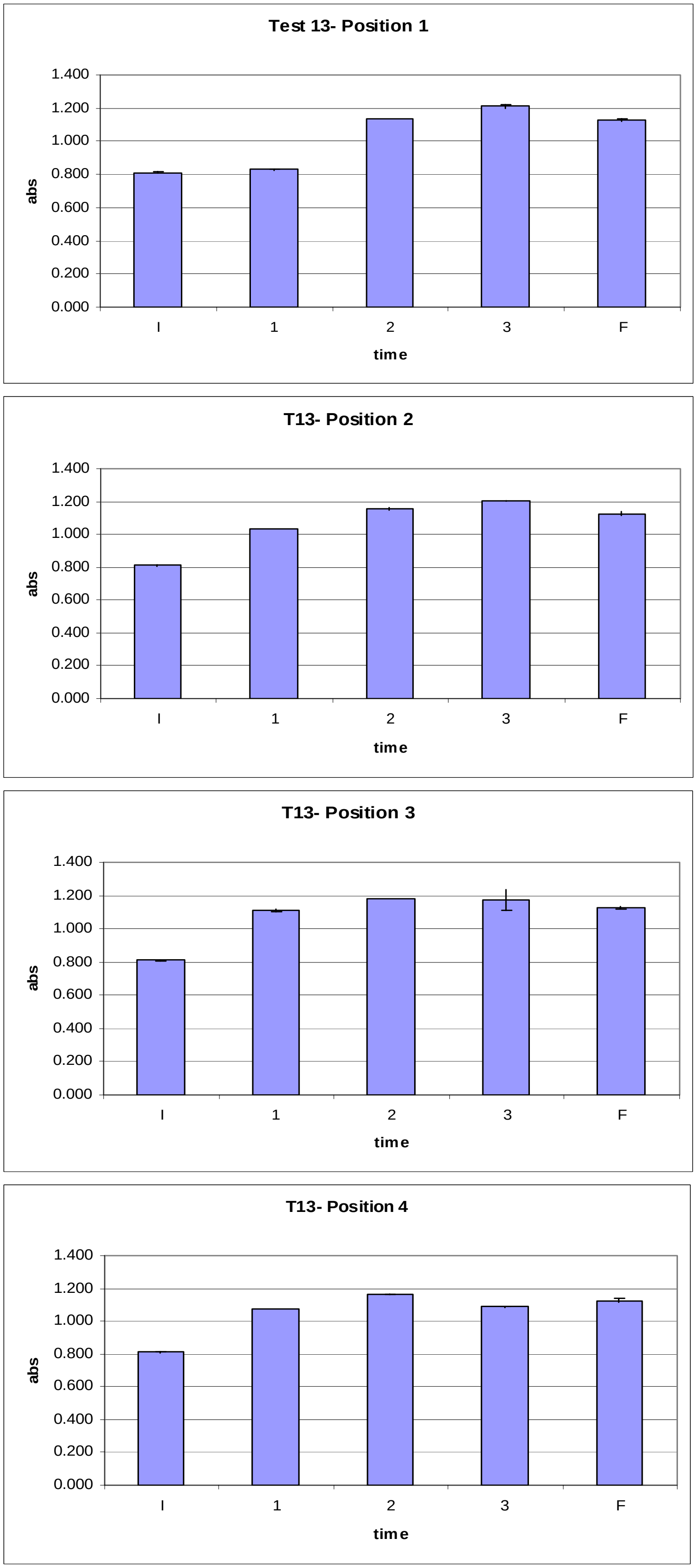


Figure 13. Test 13

Test 13 Sample	Abs	Dev
I	0.810	0.004
1-13-A1	0.831	0.005
1-13-A2	1.134	
1-13-A3	1.211	0.013
Fin	1.126	0.010
I	0.810	0.004
2-13-A1	1.038	
2-13-A2	1.16	0.01
2-13-A3	1.204	0.001
Fin	1.126	0.010
I	0.810	0.004
3-13-A1	1.111	0.007
3-13-A2	1.180	
3-13-A3	1.175	0.062
Fin	1.126	0.010
I	0.810	0.004
4-13-A1	1.077	
4-13-A2	1.164	0.002
4-13-A3	1.090	0.004
Fin	1.126	0.010

Table 13. Test 13

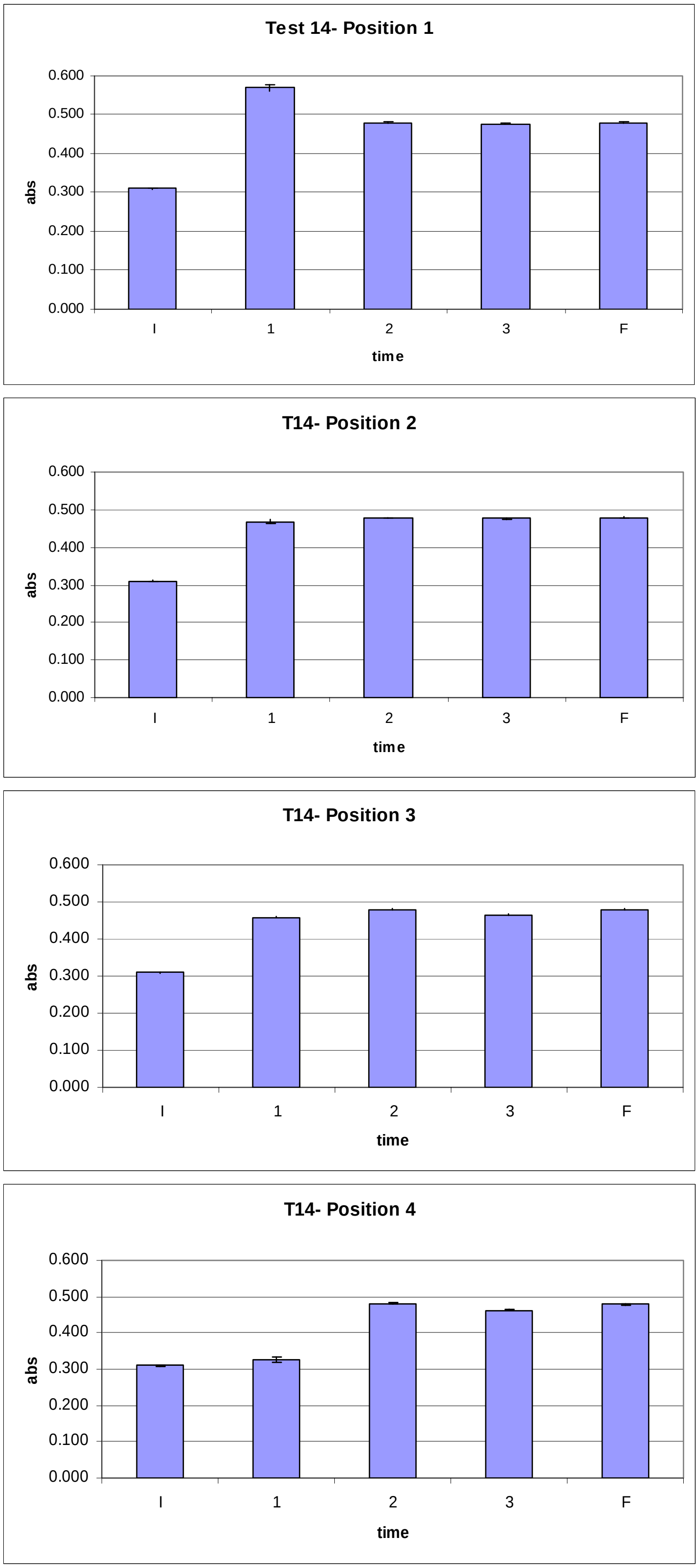


Figure 14. Test 14

Test 14 Sample	Abs	Dev
I	0.310	0.001
1-13-A1	0.569	0.008
1-13-A2	0.480	0.001
1-13-A3	0.476	0.002
Fin	0.480	0.002
I	0.310	0.001
2-13-A1	0.468	0.006
2-13-A2	0.477	0.000
2-13-A3	0.476	0.002
Fin	0.480	0.002
I	0.310	0.001
3-13-A1	0.459	0.001
3-13-A2	0.480	0.002
3-13-A3	0.465	0.002
Fin	0.480	0.002
I	0.310	0.001
4-13-A1	0.327	0.007
4-13-A2	0.481	0.002
4-13-A3	0.462	0.002
Fin	0.480	0.002

Table 14. Test 14

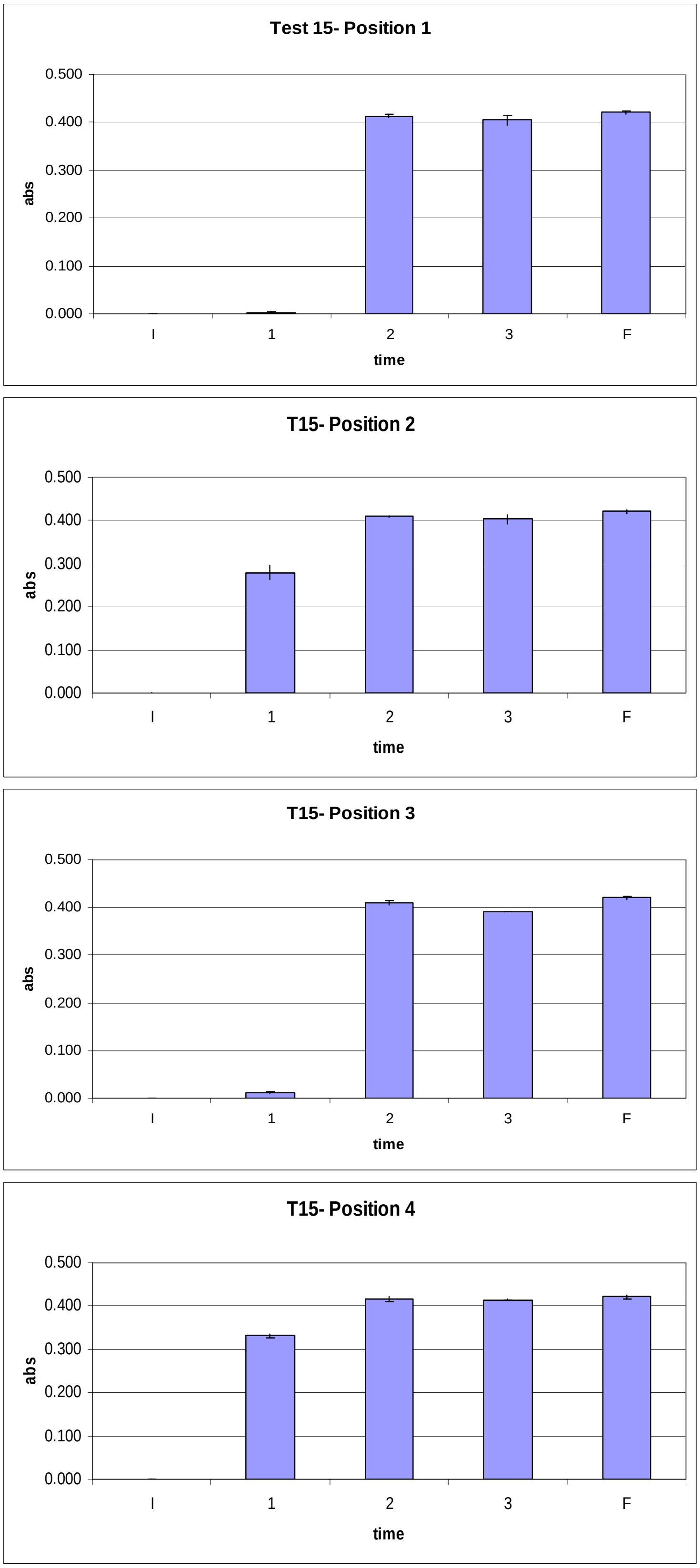
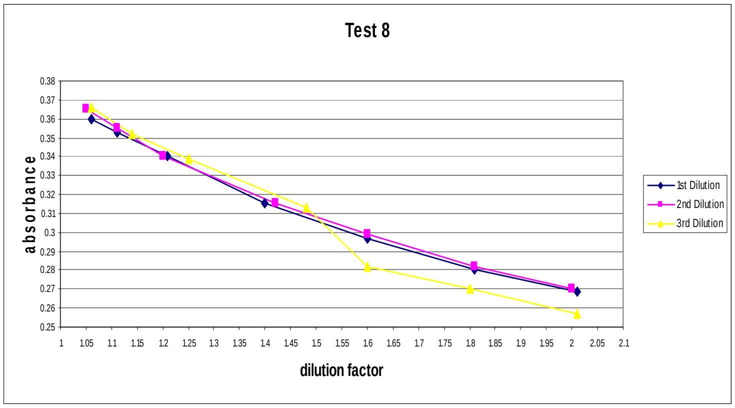
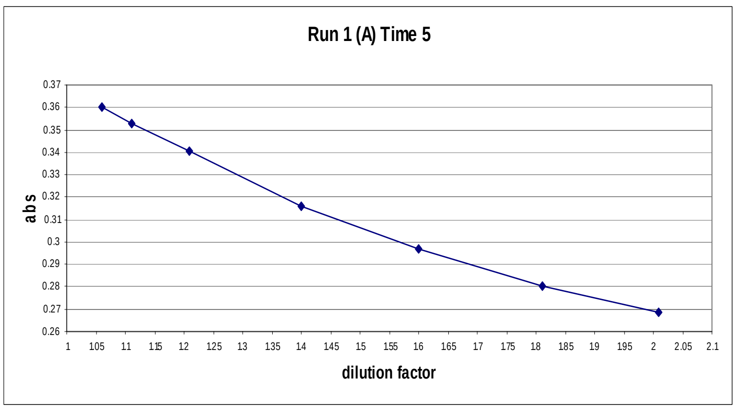


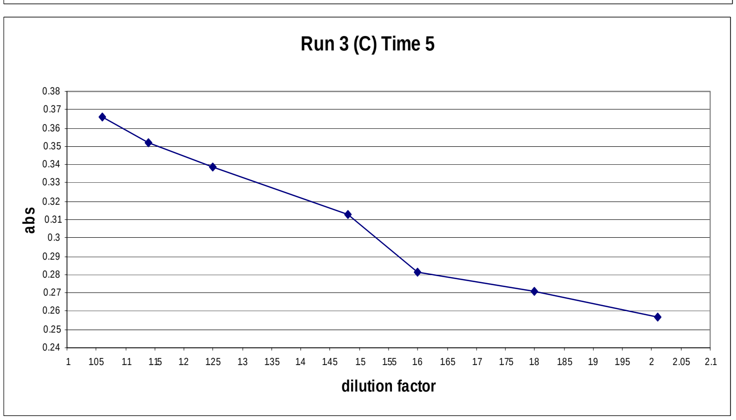
Figure 15. Test 15

Test 15		
Sample	**Abs**	**Dev**
Ini	0.0006	0.0002
1-15A-1	0.0032	0.0004
1-15A-2	0.413	0.003
1-15A-3	0.40	0.01
Fin	0.421	0.004
Ini	0.0006	0.0002
2-15A-1	0.28	0.02
2-15A-2	0.410	0.001
2-15A-3	0.40	0.01
Fin	0.421	0.004
Ini	0.0006	0.0002
3-15A-1	0.012	0.002
3-15A-2	0.409	0.005
3-15A-3	0.391	0.001
Fin	0.421	0.004
Ini	0.0006	0.0002
4-15A-1	0.331	0.005
4-15A-2	0.416	0.007
4-15A-3	0.414	0.001
Fin	0.421	0.004

Table 15. Test 15







Appendix 1. Dilution curves and mixed percent calculation

Position 1	**Abs**		**dil fac**
sample from run cond 2 (18B5)	0.279	plot on diluted 1st set	1.83
sample from run cond 3 (18C5)	0.345	plot on diluted 2nd set	1.17
DF	0.338		1.25

Run 3	1.25	1.25=100% / R3%	**80**	**% mixed**
Run 2	1.17	1.17=80% / R2%	**68**	**% mixed**
Run 1	1.83	1.83=68% / R1%	**37**	**% mixed**

Position 2	**Abs**		**dil fac**
sample from run cond 2 (28B5)	0.278	plot on diluted 1st set	1.85
sample from run cond 3 (28C5)	0.351	plot on diluted 2nd set	1.15
DF	0.338		1.25

Run 3	1.25	1.25=100% / R3%	**80**	**% mixed**
Run 2	1.15	1.15=80% / R2%	**70**	**% mixed**
Run 1	1.85	1.85=70% / R1%	**56**	**% mixed**

Position 3	**Abs**		**dil fac**
sample from run cond 2 (38B5)	0.257	plot on diluted 1st set	2.3
sample from run cond 3 (38C5)	0.348	plot on diluted 2nd set	1.16
DF	0.338		1.25

Run 3	1.25	1.25=100% / R3%	**80**	**% mixed**
Run 2	1.16	1.16=80% / R2%	**69**	**% mixed**
Run 1	2.3	2.3=69% / R1%	**30**	**% mixed**

Position 4	**Abs**		**dil fac**
sample from run cond 2 (48B5)	0.343	plot on diluted 1st set	1.19
sample from run cond 3 (48C5)	0.338	plot on diluted 2nd set	1.23
DF	0.338		1.25

Run 3	1.25	1.25=100% / R3%	**80**	**% mixed**
Run 2	1.23	1.23=80% / R2%	**65**	**% mixed**
Run 1	1.19	1.19=65% / R1%	**55**	**% mixed**

Appendix 1. (cont.) Dilution curves and mixed percent calculation