Factors Affecting Quantification of Contaminants by Image Analysis

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Abstract
Image analysis of dyed samples has become a common method of quantifying contaminants in handsheets. Five factors were tested for their impact on image analysis results. The factors were: heat drying, pressing, dye method, washing, and scanner contrast setting. Washing was the most significant factor. By improving the contrast between PSA particles and background fibers, washing tended to increase PPM values. Unfortunately, washing did not uniformly affect of the results of the two adhesive tested. Based on the results of this study an image analysis test method is proposed.

Introduction
When light strikes an object it can be scattered, transmitted, or absorbed. The amount of light scattered is related to angle of incidence, smoothness of the surface, and refractive index difference between the object and surrounding medium, typically air. Absorption is determined by the material properties of the object. If there are electronic states that can be excited by light in the visible portion of the spectrum, then some of the light intensity will be absorbed. Any light that is not scattered or absorbed is transmitted. The fate of incident light is schematically illustrated in Figure 1.

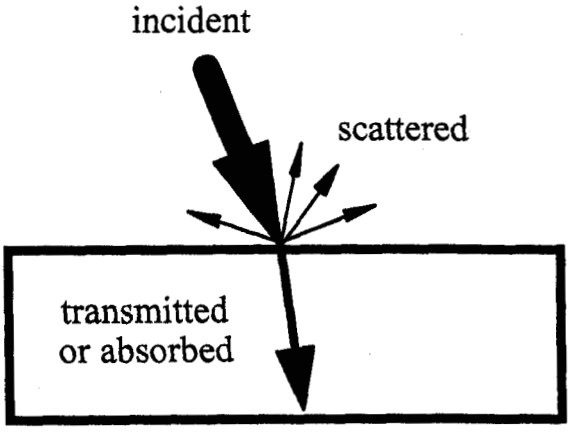

Figure 1 Illustration of the fate of light intensity when is strikes an object.

Paper is a composite of objects that can interact with incident light. The visual appearance of a sheet is the sum of individual properties of objects in the sheet, Each fiber provides surfaces for scattering and chemical constituents for absorption. When there are other objects in a sheet, e.g., dirt, pitch, stickies, etc., they can be visually identified only if they differ from fibers in their scattering or absorption properties. Since contaminants have a significant impact on quality and runnability in a recycling operation, quantifying contaminant levels is a major concern of papermakers.

A method for quantifying dirt levels in paper was codified in 1936 [1]. This method involved an operator comparing dirt particles to black dots on a standard card. By definition, dirt is any material that has sufficient contrast with the background fibers to be identified. Although attempts to computerize the method began in the 1980s [2,3], the practice of visually determining dirt counts continued, largely unchanged. Recent improvements in scanner technology have led to rapid adoption of computer image analysis-based methods by paper companies and research laboratories.

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Scanners have been common in the printing and graphics arts industries for many years. Typical prices of professional drum scanners are approximately $20-50,000. More recently developed flat bead scanners allow a sample to be placed on a glass plate as the detector assembly moves under the sample to capture the image. Due to innovations, prices of typical flat bed scanners have fallen below $1,000 and typical resolutions have increased to 2400 dots per inch, which corresponds to an image element every 10 µm. Although developed for the consumer market, some flat bed scanners have become reliable scientific instruments.

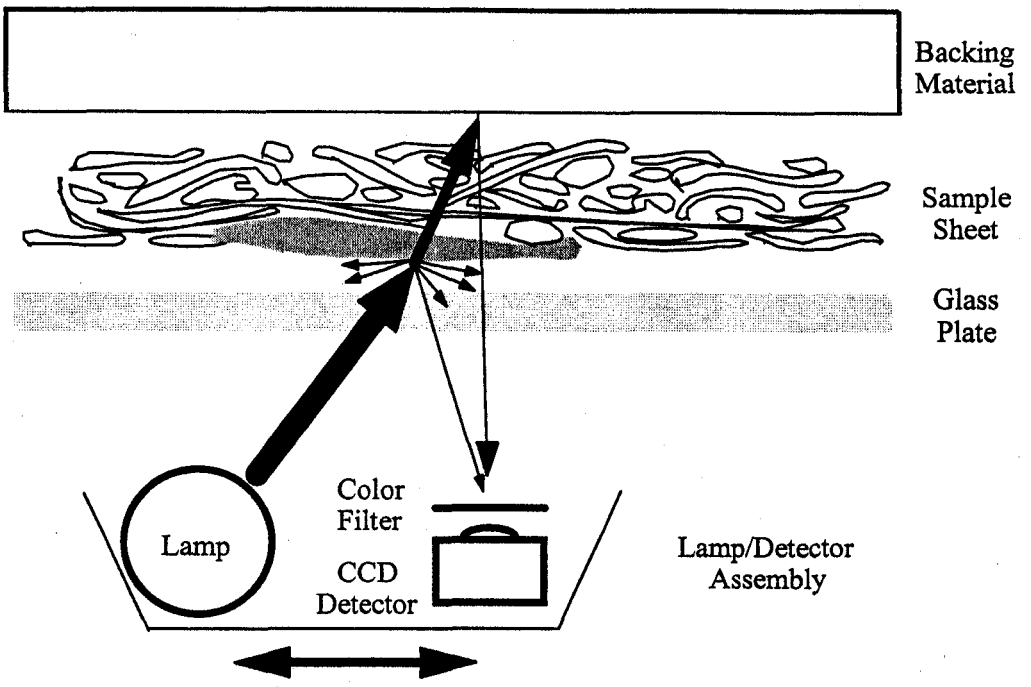

Figure 2: Typical flat-bed scanner geometry

Figure 2 shows the relative orientation of the sample, light source and detector in a representative flat bed scanner. The sample is represented as a cross section through many individual fibers. Each of these fibers is a potential site for scattering or absorption. The backing material is generally a diffuse reflecting surface that scatters light back into the sheet. The lamp is a fluorescent tube that spans the width of the scanner bed. The charge-coupled device (CCD) detector is a linear position sensitive array that also spans the width of the bed. The detector collects information from the sheet one line at a time. After a line of information is transferred, the lamp/detector assembly is moved to collect the next line of the image. In color scanners, there are three separate detectors, each with a different color filter.

As people working in the paper industry gained experience with scanners and image analysis software, they began attempting to quantify non-contrasting contaminants in paper. Important examples of such materials are pressure sensitive adhesives (PSA), waxes, and hot melt adhesives. All of these contaminants are based on polymers that are largely hydrophobic, so a common strategy is to use dyes to develop contrast with respect to cellulose fibers. A hydrophilic dye can be used to associate with fibers [3] or a hydrophobic dye can be used to associate with contaminants [4].

Recently, a method that exploits differences in refractive index has been developed [5]. The "Wet Specimen" method uses a wet sample applied to a black background to identify contaminants. Since pulp and water have similar refractive indices, wet bleached pulp fibers scatter significantly less light than dry fibers. Adhesive and wax particles, on the other hand, have refractive indices that are different than water, and so they still scatter light. Thus, a wet handsheet on a black background appears gray with white

contaminant particles. The contrast between the gray and white is generally sufficient to allow analysis with a flat bed scanner.

For image analysis of dyed samples to provide reliable quantitation of contaminants one must consider handsheet preparation, dying procedure, and operation of the scanner. The purpose of this work is to quantify the effects of heat drying, pressing, dye method, washing, and scanner contrast setting on the results of image analysis. As a consequence of these studies, an optimum test method is proposed.

Experimental Design and Methods

The trial was organized as a 2-level, 5-factor design for each adhesive tested. The adhesives were chosen to represent a range of surface properties. Since higher order interactions may be significant, a full matrix was used. The trial was conducted using a Voith 5-kg hydropulper at a 5% loading of label stock. All of the handsheets for a particular adhesive were made on the same day. A total of 16 sets of handsheets were generated from one pulper batch for each adhesive. The handsheets were generated from 10 dolar tanks. As each sheet was generated, it was labeled with a number identifying the dolar tank number and sheet number and placed on one of the 16 trial stacks. Since the stickies levels in the sheets can change as the dolar tank is drained, the first sheet of each dolar tank was added to a different trial stack each time. Each time a subsequent sheet was made it was added to the next trial stack. The end result was that each trial stack has one sheet from each dolar tank, and there was one first sheet, one second sheet, one third sheet, etc. The washing procedure was to dip the sheet in methanol, blot to remove the excess and hang to dry. A total of two washing steps were used, which gave handsheets a faint blue tint. Each set of sheets was scanned twice, once at a contrast of 20% and once at a contrast of 15%. Percent contrast was used in this study since half of the sheets were not washed, which leaves some sheets with a significant blue background.

The design is summarized in Table 1. Essentially, 16 different methods were used to process the handsheets. The key in Table 2 can be used to translate from the code in Table 1 to a particular method. For example, trial 1 was air-dried with no pressing, dyed by wiping, and not washed.

Table 1:2-Level, 4-Factor Experimental Design
(note: the fifth factor is the contrast level of the scanner)

Index	Heat	Press	Dip	Wash
	1	2	3	4
1	-	-	-	-
2	+	-	-	-
3	-	+	-	-
4	+	+	-	-
5	-	-	+	-
6	+	-	+	-
7	-	+	+	-
8	+	+	+	-
9	-	-	-	+
10	+	-	-	+
11	-	+	-	+
12	+	+	-	+
13	-	-	+	+
14	+	-	+	+
15	-	+	+	+
16	+	+	+	+

Table 2: Factors in the Experimental Design

Factor	Ident.	Description
1	Heat	- = air-dried in racks with metal disks + = heat-dried between blotter papers at 150°C for 3 min.
2	Press	- = no pressing + = one 3-minute pressing
3	Dip	- = dyeing by wiping back of blotter with Morplas Blue in heptane + = dyeing by dipping in Keystone solution in toluene/isopropanol
4	Wssh	- = no washing + = washing 2 times in methanol
5	Contrast	- = 15% contrast + = 20% contrast

Results

Image analysis was used on all handsheets. PPM, counts and particle sizes were determined for each set of sheets. Probability plotting was used to remove any handsheets that did not appear to be consistent with the rest of the set. The full data sets are included in the appendix. Since each of the five factors were varied systematically, it is possible to reliably determine the effect on the results of each factor.

The response for each factor was be determined using the Yates Algorithm[6]. The results of the analysis are presented as both the effect that a factor has on a measurement and the percentage change. Probability plotting was used to determine which factors have effects that can be discerned from the random noise in the measurements at grater than a 95% significance level. As a second check of the model, experimental response was compared to predicted response. In all cases, the residual between the data and the model exhibited a normal distribution of values, which suggests that the models were fitting all available information.

Table 3 shows the analysis of the effects of the factors on PPM values. The importance of Factor 5 (scanner contrast) is to be expected. For both adhesives, the magnitude is approximately the same and has a negative sign. Generally, when threshold value is increased, the apparent particle area increases. Factor 4 (washing) appears as a significant factor for both adhesives. The increase of PPM due to washing is likely caused by the removal of dye from the handsheet fibers. Removing dye from the fibers allows for particles deeper into the sheet to be quantified. Washing seems to increase the PPM values for Adhesive 1 more than Adhesive 2. These experiments suggest that the small particles characteristic of Adhesive 2 tend to lose contrast more quickly than those of Adhesive 1. If one assumes that the increase in PPM due to quantification of particles deeper into the sheet is the +2400 PPM as exhibited by Adhesive 1, then washing of dye from Adhesive 2 particles is likely removing the equivalent of -1470 PPM. This suggests that washing in methanol only two times results in underreporting of the Adhesive 2 loading by 14%.

Table 3: Factors that affect PPM Values

Adhesive 2			Adhesive 1		
Mean Value = 10819			Mean Value = 11674		
Factor	Effect	% Effect	Factor	Effect	%Effect
5	-1695	-16%	4	2399	21%
34	1363	13%	5	-1424	-12%
4	932	9%	34	1061	9%
1	-458	-4%	14	297	3%
2	445	4%	2	265	2%

The Yates method allows for the identification of interactions among factors. The appearance of Factor 34 (dye method x washing), suggests that dyeing with Keystone by dipping followed by washing is similar to

the wiping method without washing. This result is further supported by the fact that Factor 3 (dye method) does not appear as a significant factor for either adhesive.

Factor 1 (heat-drying) only seems to negatively affect PPM value for Adhesive 2. There seems to be a loss Of 4%, which could be due to particle contraction or to evaporation of some low molecular weight component of the adhesive during drying.

Analysis of the PPM standard deviation of the sheets in the sets showed that for both adhesives there was no significant effects. This result suggests that none of the factors affect repeatability.

Table 4: Factors that affect Particle Sizes

Adhesive 2			Adhesive 1		
Mean Value = 0.285 mm^2			Mean Value = 0.935 mm^2		
Factor	Effect	%Effect	Factor	Effect	%Effect
4	0.038	13%	4	0.275	29%
34	0.020	7%	34	0.105	11%
5	-0.015	-5%	5	-0.108	-12%
3	-0.014	-5%	3	-0.086	-9%
14	0.014	5%	45	-0.060	-6%
13	-0.013	-4%			
134	-0.010	-3%			
1	0.008	3%			

Table 4 shows the factors that significantly affect particle size. The major factors were identified for PPM values, Table 3. Washing has the largest impact on size for both adhesives. Washing increases apparent area by increasing the contrast with surrounding fibers. As with PPM, Factor 34 is significant. This result suggests that dying with Keystone and washing give similar results to dyeing with Morplas and not washing. The appearance of Factor 3 suggests that the Keystone dye maybe slightly better bound to the particles than the Morplas dye. Finally, Factor 1 (heat drying) seems to have a small effect on particle size. However, since several factors interact with it, 14, 13, and 134, a clear conclusion cannot be made.

Table 5: Factors that affect Particle Count

Adhesive 2			Adhesive 1		
Mean Value=683/sheet			Mean Value 228/sheet		
Factor	Effect	% Effect	Factor	Effect	% Effect
5	-69	-10%	3	22	10%
134	46	7%	4	-20	-9%
3	45	7%	34	-12	-5%
1	-37	-5%	1	10	4%
34	33	5%	45	9	4%
2	31	5%			
13	27	4%			
4	-28	-4%			
24	23	3%			

Table 5 shows the factors that affect particle counts. The factors have a lower impact on counts than either PPM or particle size. This insensitivity suggests that the number of counts may be a better measurement than PPM. The appearance of Factor 5 for Adhesive 2 may indicate that the particles of this adhesive are not stained very darkly and their corresponding gray values are near the threshold. This indicates a

potential problem. Generally, threshold values are chosen to make the number of counts insensitive to threshold.

The fact that Factor 4 (washing) has a positive impact on particle size and a negative impact on particle count suggests that washing is allowing for particles to be more completely quantified. It is possible that with low contrast to the background, a single large particle will appear to be broken into several smaller ones by overlying fibers. After washing, the contrast becomes sufficient for the software to correctly reconnect these pieces.

Finally, since sheet consolidation affects the amount of scattering by the fibers of the sheets, it can have an impact on image analysis results. The caliper of the sheets was measured. Table 6 shows the factors that affect sheet caliper. Not surprisingly, pressing the sheet is the only factor that has a major effect. Since pressing has only a 2-4% impact on PPM values, one can conclude that sheet consolidation has a minor impact on image analysis results.

Table 6: Factors that affect Sheet Caliper

Mean Value = 0.1426 mm		
Factor	Effect	% Effect
2	-0.0189	-13%
1	0.0021	1%
12	0.0015	1%

Conclusions

Factor 4 (washing) appears to be the single most significant factor for all measurements. Washing dye from the fibers in the sheet seems to allow for particles to be more completely quantified. Washing does not affect both adhesives equally. Two washings in methanol reduces the PPM values for Adhesive 2 by 14%.

Factor 1 (heat-drying) does not affect the results for Adhesive 1. For Adhesive 2, heating has a complex effect that interacts with several other factors. The data do show, however, that the particle size is only slightly changed < 4%, which rules out any significant heat induced flow.

Factor 3 (dyeing method) does not have a major impact on any of the results. It only appears as an interaction with Factor 4 (washing).

Of the three major measurements, PPM, particle size, and particle counts, the one least affected by variations in the sheet preparation methods is particle counts.

Recommendations

A proposed test method is shown below. Since washing is a major factor, the inclusion of washing in a test method is problematic. Furthermore, the results here suggest that the effect of washing is not the same for both adhesives. Since the test method should be insensitive to adhesive chemistry, particle size and particle morphology, the suggested test method does not include a washing step.

Finally, heat-drying the sheets does not seem to induce large changes in particle size for the adhesives used in this study. Given the significant time advantages of heat-drying over air-drying, the new test method uses heat-drying, but further testing with other adhesive types is likely necessary to be assured that heating does not affect results.

Hydrophobic contaminant identification method

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1. Scope

1.1 This procedure describes a method for quantifying hydrophobic contaminants in pulp samples. A dye that associates with contaminant particles is used to develop contrast from the pulp background. Computer-based color image analysis can then be used to quantify contamination levels by type based upon color and shade.

1.2 This method is a compliment to TAPPI T 213 "dirt in pulp". It allows for the quantification of hydrophobic contaminants that do not have sufficient contrast with pulp to be identified. Hydrophobic contaminants, i.e., waxes, pressure sensitive adhesives, hot melt adhesives, etc., can contribute to "stickie" problems in recycled fiber mills.

2. Significance

2.1 When coupled with automated color image analysis contaminant measurement and classification methods, this method provides a more complete quantification of pulp contaminants.

3. Apparatus

3.1 Standard handsheet mold as described in TAPPI T 205 sp-95.
3.2 Standard couch roll.
3.3 Standard blotting paper as described in TAPPI T 205 sp-95.
3.4 Handsheet dryer with an operating temperature of 150 °C.
3.5 Image analysis system.
3.6 Plastic or metal tray large enough to hold a blotting paper.
3.7 Foam varnish applicator.
3.8 Laboratory timer
3.9 Standard press as described in TAPPI T 205 sp-95

4. Reagent

4.1 dye solution 0.67 g of C.I. solvent blue 36 in 1 liter of n-heptane. The dye is also known as Morplas Blue 1003 and can be obtained from Pylam Products Company Inc., 2175 East Cedar Street, Tempe, AZ 8528 1, (602) 929-0070

5. Procedure

5.1 Form handsheets according to TAPPI T 205, except end the procedure after couching. After couching the second wet blotting paper is discarded and a third dry one is placed to protect the handsheet attached to the first blotting paper.

5.2 Stack handsheets and blotters, using care to align sheets in a uniform stack. Place the stack in a press and, over a period of 30 seconds, raise the pressure to 345 kPa (50 psig). Maintain this pressure for 5 minutes.

5.3 Carefully unstack the sheets and place each pair of blotter papers, with a handsheet between them, on the dryer. The intent is to leave the handsheet firmly attached to the couching blotting paper until it is dyed. Dry for 3 minutes at 150°C with gentle restraint.

5.4 Dye the handsheet by applying the dye solution to the back side of the blotter that has a handsheet attached. This allows the dye to uniformly penetrate the handsheet. Furthermore, as the dye solution passes through the blotting paper undissolved dye particles are filtered from the solution. Typically, dying is done by placing the blotting paper/handsheet with the handsheet side down on another blotting paper in a tray, and then painting the blotting paper with a foam brush that has been dipped in the dye solution, This step of the procedure should be carried-out in a ventilation hood to avoid exposure to heptane vapors. Since the dye is a mild sensitizer, heptane-tolerant gloves are also required.

5.5 Let the heptane evaporate from the blotting paper/handsheets without separating the handsheet from the blotter paper by hanging them with clips attached to the blotter paper in the ventilation hood. Typical drying times are 2-3 minutes.

5.6 With a gloved hand, gently peel the dyed handsheet from the blotting paper. Place the side that was towards the blotting paper on the glass of the flat bed scanner. Using a weight with a white surface, hold the sheet flat on the scanner.

5.7 Use the color image analysis software to quantify the number of particles on the sheet. To compensate for sheet to sheet variations in dye intensity, best results are obtained by using a threshold that is automatically set 20% below the mode of the sheet image picture point luminance value frequency distribution. The software system developed by Verity IA LLC was used in this study. The scanner used was an AGFA Argus II.

6. Report

6.1 Results are reported as parts per million of the scanned area. Typically an average and 95% confidence interval for 10-40 standard 1.2 g handsheets is calculated.

References

[1] TAPPI Test Method T 2 13 om-97.

[2] R. Trepanier et al. TAPPI Journal Dec 1989 p. 153.

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[4] C. Houtman et al. Proceedings of the TAPPI 2000 Recycling Conference, Vol. 2 p.403.

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[6] G.E.P Box et al. "Statistics for Experimenters" John Wiley and Sons, NY, 1978, p. 323.

Table 7: Data values for Adhesive 1

Design						PPM	StDev	Count	Size	Caliper
	1	2	3	4	5			cnt/sheet	mm^2	mm
1	-	-	-	-	-	12024	1565	202	1.033	0.157
2	+	-	-	-	-	11431	848	231	0.874	0.158
3	-	+	-	-	-	11685	1427	201	1.040	0.119
4	+	+	-	-	-	11594	1540	247	0.807	0.124
5	-	-	+	-	-	9172	1572	302	0.522	0.159
6	+	-	+	-	-	8479	1508	305	0.472	0.165
7	-	+	+	-	-	10714	2988	295	0.385	0.120
8	+	+	+	-	-	9298	1493	308	0.526	0.130
9	-	-	-	+	-	13556	1640	181	1.339	0.164
10	+	-	-	+	-	14913	1399	200	1.285	0.163
11	-	+	-	+	-	15008	1685	185	1.487	0.119
12	+	+	-	+	-	15353	1418	203	1.318	0.129
13	-	-	+	+	-	15191	1225	205	1.359	0.163
14	+	-	+	+	-	17394	844	227	1.334	0.163
15	-	+	+	+	-	17140	1580	193	1.537	0.122
16	+	+	+	+	-	16611	3127	217	1.368	0.125
17	-	-	-	-	+	9879	1297	200	0.853	0.157
18	+	-	-	-	+	8848	815	210	0.763	0.158
19	-	+	-	-	+	9322	1273	208	0.719	0.119
20	+	+	-	-	+	9144	1373	220	0.713	0.124
21	-	-	+	-	+	6848	1185	243	0.505	0.159
22	+	-	+	+	+	6123	1064	262	0.447	0.165
23	-	+	+	-	+	6940	1469	263	0.464	0.120
24	+	+	+	-	+	6893	930	277	0.430	0.130
25	-	-	-	+	+	11046	1388	191	1.000	0.164
26	+	-	-	+	+	11589	1252	214	0.948	0.163
27	-	+	-	+	+	11664	640	194	1.130	0.119
28	+	+	-	+	+	12216	1126	211	1.025	0.129
29	-	-	+	+	+	12470	1275	211	1.107	0.163
30	+	-	+	+	+	13581	956	240	1.022	0.163
31	-	+	+	+	+	14150	2208	209	1.092	0.122
32	+	+	+	+	+	13293	2269	235	1.011	0.125

Table 8: Data values for Adhesive 2

Design						PPM	StDev	Count	Size
	1	2	3	4	5			cnt/sheet	mm^2
1	-	-	-	-	-	12589	2180	764	0.301
2	+	-	-	-	-	11092	1337	717	0.280
3	-	+	-	-	-	13560	1144	790	0.300
4	+	+	-	-	-	12832	942	800	0.301
5	-	-	+	-	-	11522	1064	872	0.249
6	+	-	+	-	-	9961	965	785	0.202
7	-	+	+	-	-	10984	1095	847	0.221
8	+	+	+	-	-	8810	1122	726	0.226
9	-	-	-	+	-	11548	1099	663	0.287
10	+	-	-	+	-	10021	1675	461	0.372
11	-	+	-	+	-	12897	1160	804	0.289
12	+	+	-	+	-	12984	1278	572	0.392
13	-	-	+	+	-	14956	2370	718	0.351
14	+	-	+	+	-	14490	742	771	0.350
15	-	+	+	+	-	15841	1991	811	0.340
16	+	+	+	+	-	16141	938	937	0.333
17	-	-	-	-	+	9803	1188	629	0.264
18	+	-	-	-	+	8090	1091	576	0.259
19	-	+	-	-	+	10058	1011	655	0.266
20	+	+	-	-	+	10106	1017	662	0.274
21	-	-	+	-	+	8262	862	662	0.213
22	+	-	+	-	+	6548	667	613	0.192
23	-	+	+	-	+	7769	1178	703	0.200
24	+	+	+	-	+	6221	772	566	0.198
25	-	-	-	+	+	8901	680	629	0.252
26	+	-	-	+	+	6437	1159	356	0.340
27	-	+	-	+	+	9504	1035	680	0.262
28	+	+	-	+	+	8932	731	455	0.343
29	-	-	+	+	+	11053	1955	595	0.311
30	+	-	+	+	+	10722	628	612	0.320
31	-	+	+	+	+	11193	438	691	0.318
32	+	+	+	+	+	12393	1327	728	0.317