DEVELOPMENT OF USPS LABORATORY AND PILOT-SCALE TESTING PROTOCOLS

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ABSTRACT

The ultimate goal of the US Postal Service (USPS) Environmentally Benign Stamp Program is to develop stamp adhesives that can be removed by unit operations found in recycling mills. The maintenance of final product quality specifications for a recycling mill while loading the feedstock with a significant quantity of adhesive is the criterion for success of this program. However, since it is neither prudent nor cost-effective to test all experimental adhesive materials at mill-scale, we have developed laboratory-scale (360 g pulp) and pilot-scale (112 kg pulp) protocols for testing adhesive performance in recycling environments. furthermore, since adhesive quantification methods are critical to testing on laboratory, pilot and mill-scales, a computer image analysis protocol, which uses hydrophobic dyes to provide contrast between fiber and adhesive particles, has also been developed.

INTRODUCTION

Before initiation of the USPS Environmentally Benign Stamp Program, no widely accepted protocol existed for determining the compatibility of pressure sensitive adhesives (PSAs) with paper recycling processes. Thus, there was no unified guidance for adhesive formulators in their efforts to develop new products in response to environmental concerns. The current protocols, which are included as appendices, are the product of several years of testing experimental adhesives submitted by formulators. The results of these recycling tests and performance tests, described elsewhere in these proceedings, have been used to eliminate adhesives that are unlikely to meet all of the specifications for new stamp materials. Thus the large effort required to take an adhesive to a mill trial was only expended on adhesives that have a high likelihood of success.

During the development of these protocols, several operators of recycling mills gave suggestions of operating parameters and process sequence. We drafted a protocol detailing process configurations and operating parameters that represent an average process. This proposal was reviewed and approved by our cooperators. Although the results from laboratory and pilot-scale experiments must be interpreted using extrapolations and approximations, the participating mill operators have indicated these data allow them to estimate the performance of an adhesive in their mill. Furthermore, experiments conducted using these protocols do provide a uniform basis of comparing different adhesive formulations.

The two unit operations that are most effective for removing adhesive from pulp are at the center of the laboratory and pilot-scale protocols, screening and flotation. The balance of this report will be devoted to discussing the two testing protocols.

The use of trade, firm or product names is for reader information and does not imply endorsement by the U.S. Department of Agriculture or the Forest Service.

LABORATORY-SCALE PROTOCOL

The laboratory-scale protocol consists of 4 major unit operations and two dewatering operations. These unit operations are shown in Figure 1, a flowsheet for the laboratory-scale testing. The details of the protocol are given in Appendix 1. The first operation is high-consistancy, 15 %, pulping of 360 g OD pulp which contains 1 % adhesive by weight. After 0.3 and 0.15 mm slotted screening with a Valley Flat Screen, the pulp slurry is further cleaned by flotation in a Denver Flotation device. Four pulp samples are taken: after pulping, after 0.3 mm screening, after 0.15 mm screening, and after flotation. These samples are analyzed for residual adhesive levels using the USPS Image Analysis protocol. The two screening and flotation reject samples are dried and weighed. Visual inspection is used to estimate the fraction of fiber in these samples, which allows for the estimate of the mass of adhesive removed in each unit operation.

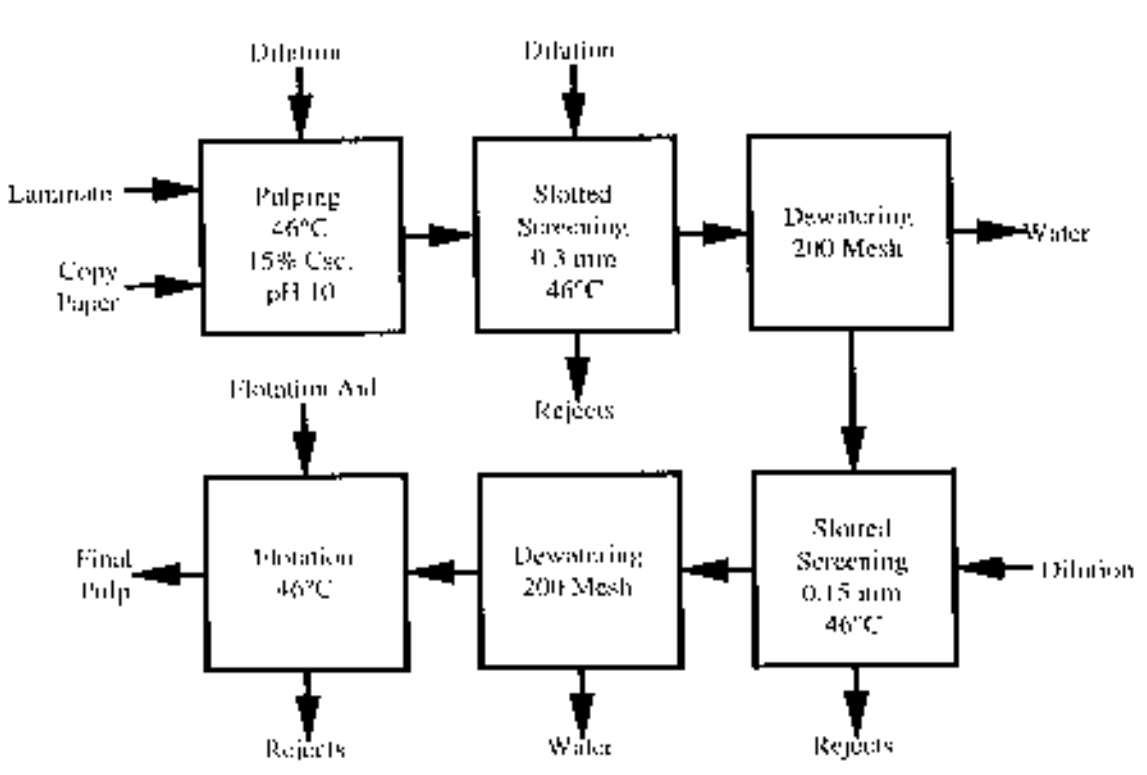

Figure 1: Flowsheet for the laboratory-scale protocol.

A major advantage of the laboratory-scale protocol is that it only requires 4 g of adhesive. Thus it can be used to test a large number of adhesive formulations relatively rapidly, which should aid in future development of new adhesives. Once promising candidates are found they are submitted for more thorough testing at pilot and mill-scales.

PILOT-SCALE PROTOCOL

Figure 2 shows the flowsheet for the USDA Forest Products Laboratory (FPL,) pilot plant. The details of the protocol are described in Appendix 2. Essentially, this protocol provides six major unit operations: pulping, slotted screening, forward cleaning, flow-through cleaning, flotation and washing.

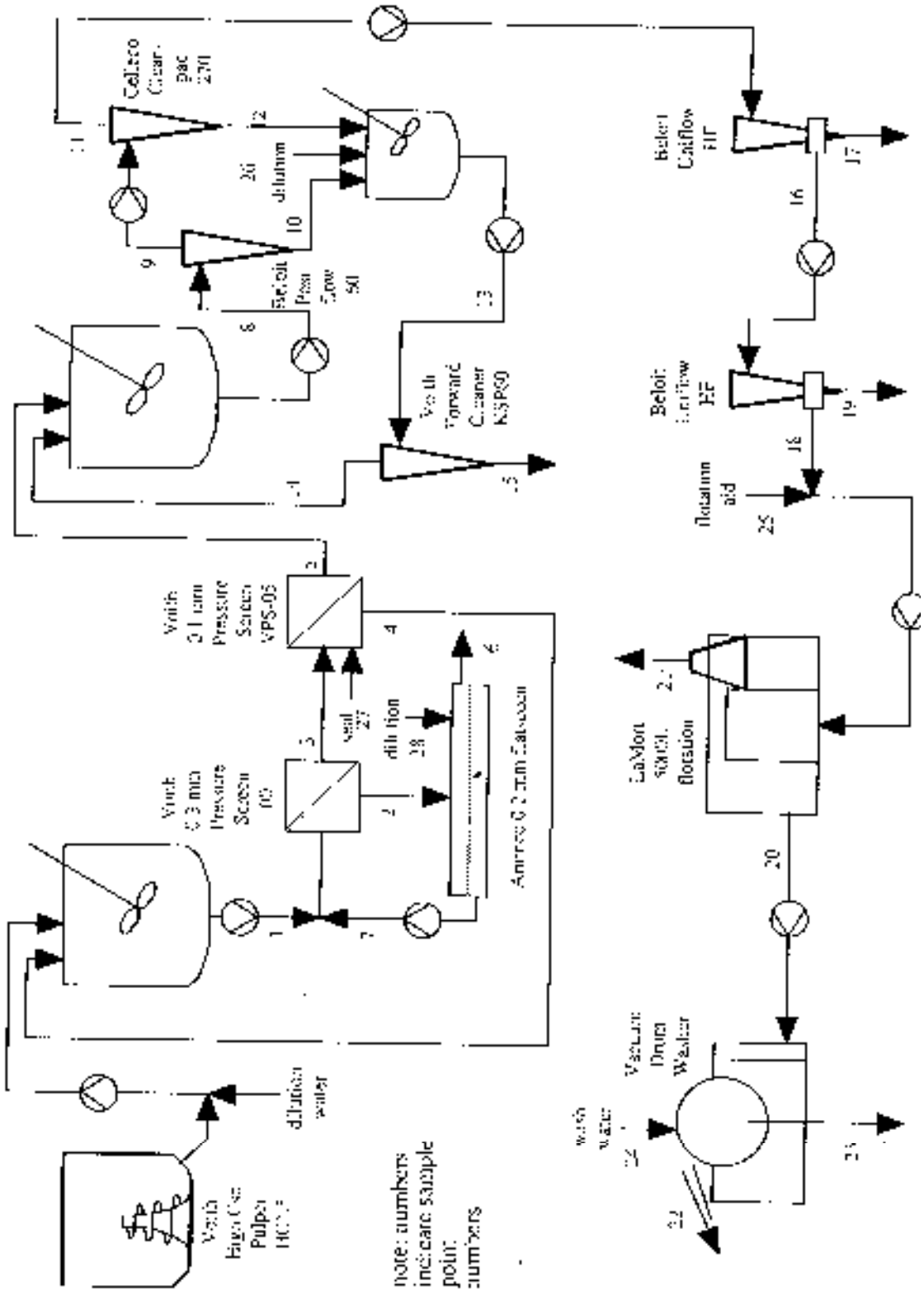

Figure 2 : FPL Pilot Plant Flowsheet

Although adhesive removal efficiencies from experiments using flat screens can be indicative of product performance, experiments with pressure screens better represent mill-scale operations. Furthermore, since slotted screening will be essential to removing most adhesive formulations, the pilot protocol was developed around commercial pressure screens. The sizes and flowrates in the protocol were set to give approximately 1 hour of screen operation. The screen sizes, consistencies and passing speeds are shown in Table 1. Typical pressure screen passing speeds in recycling mills are 0.6 to 2 m/s. Unfortunately, due to down-stream limitations, the passing speeds for the pilot plant trials were 0.35-0.4 m/s. Lower passing speeds are often associated with higher screening efficiencies, at a cost of fiber fractionation.

Table 1 : Pressure Screen Operating Parameters

Slot Size	Inlet Csc.	Open Area	Passing Speed
0.3 mm milled	1.10 %	4.3 %	0.41 m/s
0.1 mm profile	0.94 %	2.3 %	0.35 m/s

As is shown in the flowsheet, Figure 2, rejects from the 0.3-mm primary pressure screen are sent to a 0.2.-mm flat screen. Accepts from this screen are returned to the inlet of the 0.3-mm primary screen in a cascade back configuration. The rejects from the 0.1-mm pressure screen are returned to the stock tank, without further processing. Unfortunately, since approximately 30% of the stock remains in the pressure screen tank at the end of the run, this configuration makes direct calculation of the fiber yield difficult. A dynamic mass balance calculation suggests that 57% of the stock in the pressure screen feed tank are rejects from the 0.1-mm screen at the end of a typical run. Using this percentage, the pressure screen rejects remaining in the tank were estimated to be 24.3 kg. Since the total fiber rejected by the 0.1-mm screen was 42 kg, approximately 57% of the rejects were recovered during the run. This recovery rate is likely significantly lower than that of a typical mill-scale recycling operation. The details of a yield calculation are shown in Table 2.

Table 2: Fiber yield calculations

Stock pulped		112.5 kg
Initial ash	11.5%	
Stock remaining in feed tank	42.6	
Percent stock that is rejects	57.0%	
Stock not fed to system		-18.3 kg
Screen rejects	24.3	
Forward cleaner rejects	5.9	
Flow-through cleaner rejects	3.0	
Flotation Rejects	1.6	
Washing Rejects	1.7	
Total rejects		-36.6 kg
Samples taken		-2.4 kg
Fiber recovered		54.7 kg
Final ash	1.0%	
Fiber yield		67.3 %

The total fiber yield of 67% is likely lower than a typical mill-scale operation. Significant yield improvements could be achieved by adding secondary pressure screens to the system, which is planned for the near future.

During a trial temperatures and flowrates are recorded using a computer data acquisition system. These data are used to monitor the operating parameters. Consistencies are also determined on all the process streams. These data for six trials are shown in Tables 3-5. The physical and chemical properties of adhesives are very temperature sensitive. Thus temperature must be considered the most important operating parameter. The design of the pilot plant system includes temperature controllers on all process water streams, but tank temperatures are not controlled. Table 3 shows average temperature data for six trials. The data shown in Table 3 represent an average of approximately 500 temperature readings taken during the trial. Only data for the pulping and screening sections are include as these temperatures likely have the most impact on adhesive performance. Inspection of Table 3 shows that the temperature is well controlled. Although no temperature data exist for the pressure screens, it has been estimated that screening occurs at approximately 43°C.

Table 3: Pulping and screening section temperatures

#	Description	Trial No.						Ave	SD	COV
		#244	#245	#246	#247	#251	#258			
1	Pulper Temperature	47.1	46.5	47.3	47.5	44.1	46.0	46.4	1.3	3%
2	Feed Tank Temperature	44.2	45.0	42.9	41.3	42.1	43.3	43.1	1.3	3%
3	Flat Screen Feed Water Temperature	46.0	45.9	46.0	40.4	45.9	45.9	45.0	2.2	5%
4	Accept Tank Temperature	39.6	38.4	41.3	41.2	41.5	41.7	40.6	1.3	3%
5	Ambient Temperature	21.9	24.0	27.2	25.9	22.8	20.9	23.8	2.4	10%

Note all temperatures are shown in °C.
SD = Standard deviation
COV = Coefficient of variation = 100%, x SD/Ave.

Other experimental data acquired during the trial include visual observations of the size and shape of the adhesive particles collected on the flat screen, mass of rejects collected on the flat screen, power consumption during pulping, observations about the difficulty of cleaning the tanks and equipment, amount of foam in the flotation cell, and biological oxygen demand (BOD) analysis of the wash water leaving the vacuum drum washer.

Results of adhesive removal by the FPL Pilot Plant system have been compared to those from other pilot plants and mills. These comparisons have shown that the FPL system has a higher adhesive removal efficiency than any other system. This is likely due to (1) the use of only clean water for dilution, (2) a screening system with few low passing velocities, (3) clean feed stock, and (4) thoroughly cleaning the system between runs. Thus if the pilot plant data are to be used for predicting the recycling performance of an adhesive on a mill-scale, these data should be interpreted as the best case.

IMAGE ANALYSIS PROTOCOL

PSA particles in a mill setting are often coated with colored contaminants, which can make them visible in sample handsheets. With the relatively clean pulp slurries used in the lab and pilot-scale experiments, PSA particles are often white and have low contrast with background fibers. To qualify the PSA concentration in pulp samples the particles were dyed to increase the contrast with the background fibers. For laboratory and pilot-scale experiments, handsheets were dipped in a solution of a hydrophobic dye. The dye associates strongly with any hydrophobic particles in the sample. Since PSAs are often hydrophobic, they lend to retain dye while cellulose fibers, which are hydrophilic, do not. The result of washing with methanol is a handsheet with dark blue PSA particles and a white or lightly tinted background. Once the PSA particles are made visible by dying, the amount of adhesive is quantified by using a flatbed scanner and image analysis software. The details of the USPS Image Analysis protocol are given in Appendix 3.

CONCLUSIONS

Laboratory and pilot-scale PSA testing protocols have been developed. The laboratory-scale protocol can be used to test a large number of experimental adhesives quickly. The pilot-scale testing requires more material but more completely represents the unit operations found in a typical recycling mill. Due to the cleanliness of the system and the low screen passing speeds, the pilot plant data likely represent an upper limit for the PSA removal efficiencies one might expect in a mill. The USPS Image Analysis protocol has proven useful in quantifying PSA levels in various stock samples.

Table 4: Process flowrates for the repeat trials

Strm	Description	Trial Number								
#		#244	#245	#246	#247	#251	#258	Ave	SD	COV
1	Feed to 0.3 mm p. screen	128.8	130.7	137.4	129.9	130.6	126.2	130.6	3.72	3%
2	Rejects of 0.3 mm p. screen	45.1	44.2	45.1	45.0	45.4	43.8	44.8	0.62	1%
3	Accepts of 0.3 mm p. screen	254.2	257.8	258.8	253.7	250.8	259.0	255.7	3.32	1%
4	Rejects of 0.1 mm p. screen	45.1	51.7	52.1	51.3	51.3	51.3	50.5	2.65	5%
5	Accepts of 0.1 mm p. screen	201.0	204.3	203.7	204.9	204.1	206.6	204.1	1.83	1%
2	0.2 mm flat screen feed	45.1	44.2	45.1	45.0	45.4	43.8	44.8	0.62	1%
28	Process water	146.1	150.8	147.9	151.4	147.6	139.6	147.2	4.25	3%
6	0.2 mm flat screen rejects	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01		
7	0.2 mm flat screen accepts	176.3	176.7	175.7	178.8	176.1	177.9	176.9	1.19	1%
8	Forward cleaner feed	261.2	236.3	256.5	264.6	262.1	262.9	257.3	10.63	4%
9	1st forward cleaner accepts	234.6	211.2	227.9	234.5	233.5	233.8	229.3	9.19	4%
10	1st forward cleaner rejects	26.6	25.1	28.6	30.1	28.6	29.1	28.0	1.83	7%
9	2nd forward cleaner feed	239.9	217.2	236.9	236.7	234.4	233.2	233.1	8.10	3%
11	2nd forward cleaner accepts	217.2	197.5	215.1	214.7	212.5	211.7	211.5	7.11	3%
12	2nd forward cleaner rejects	22.7	19.7	21.8	22.0	21.9	21.5	21.6	1.01	5%
13	Fiber recovery cleaner feed	74.6	67.6	72.0	72.8	74.2	73.9	72.7	2.58	4%
14	Fiber recovery cleaner accepts	67.3	59.7	64.8	63.8	65.8	66.1	64.6	2.67	4%
15	Fiber recovery cleaner rejects	10.8	9.1	9.7	10.0	9.4	9.3	9.7	0.62	6%
26	Dilution water to fiber recovery	30.1	28.5	28.2	26.6	29.6	29.5	28.8	1.27	4%
11	1st thru-flow cleaner feed	208.5	199.7	211.1	199.9	208.0	209.4	200.1	4.99	2%
16	1st thru-flow cleaner accepts	179.6	169.2	178.4	177.9	177.8	186.6	178.3	55.5	3%
17	1st thru-flow cleaner rejects	23.8	23.9	24.2	23.4	24.4	26.4	24.4	1.06	4%
16	2nd thru-flow cleaner feed	188.4	188.3	188.4	187.8	187.6	189.2	188.3	0.56	0%
18	2nd thru-flow cleaner accepts	168.3	171.8	169.7	167.4	167.2	165.5	168.3	2.19	1%
19	2nd thru-flow cleaner rejects	23.2	23.5	24.4	22.8	24.2	23.7	23.6	0.60	3%
18	Lamort flotation cell feed	168.3	171.8	169.7	167.4	167.2	172.7	169.5	2.31	1%
20	Lamort flotation cell accepts	166.1	164.6	166.3	160.8	162.8	165.7	164.4	2.18	1%
21	Lamort flotation cell rejects	2.2	7.2	3.4	6.6	4.4	4.1	4.7	1.91	41%
22	Pulp from drum washer	3.9	4.1	3.8	4.0	4.1	4.2	4.0	0.14	3%
23	Washwater from drum washer	156.5	125.1	102.0	136.2	106.0	124.4	125.0	20.04	16%

Note All flowrates have the units of Liters/minute.
SD = Standard deviation
COV = Coefficient of variation = 100% x SD/Ave.

Table 4: Process Consistencies

Strm	Description	Trial Number								
#		#244	#245	#246	#247	#251	#258	Ave	SD	COV
l(a)	Pulper sample	12.25	12.11	12.77	12.60	14.50	13.12	12.89	0.8672	7%
1	Feed to 0.3 mm p. screen	2.241	2.312	2.107	2.052	2.150	2.257	2.187	0.0995	5%
2	Rejects of 0.3 mm p. screen	1.249	1.383	1.261	1.174	1.268	1.347	1.280	0.0746	6%
3	Accepts of 0.3 mm p. screen	0.917	0.990	0.938	0.933	0.024	0.971	0.945	0.0286	3%
4	Rejects of 0.1 mm p. screen	1.794	2.047	1.817	2.005	1.840	1.866	1.895	0.1050	6%
5	Accepts of 0.1 mm p. screen	0.742	0.766	0.682	0.736	0.732	0.745	0.734	0.0280	4%
2	0.2 mm flat screen feed	1.249	1.383	1.261	1.174	1.268	1.347	1.280	0.0746	6%
28	Process water	-	-	-	-	-	-			
6	0.2 mm flat screen rejects	-	-	-	-	-	-			
7	0.2 mm flat screen accepts	0.272	0.335	0.332	0.323	0.273	0.292	0.305	0.0289	10%
8	Forward cleaner feed	0.604	0.627	0.617	0.647	0.613	0.618	0.621	0.0147	2%
9	1st forward cleaner accepts	0.485	0.522	0.494	0.530	0.483	0.521	0.506	0.0206	4%
10	1st forward cleaner rejects	1.602	1.896	1.146	1.796	1.593	1.248	1.547	0.2962	19%
9	2nd forward cleaner feed	0.485	0.522	0.494	0.530	0.483	0.521	0.506	0.0206	4%
11	2nd forward cleaner accepts	0.457	0.490	0.482	0.491	0.493	0.480	0.484	0.0149	3%
12	2nd forward cleaner rejects	0.603	0.648	0578	0.651	0.614	0.623	0.619	0.0276	4%
13	Fiber recovery cleaner feed	0.679	0.747	0.697	0.692	0.712	0.672	0.700	0.0271	4%
14	Fiber recovery cleaner accepts	0.6.58	0.607	0.665	0.646	0.700	0.623	0.665	0.0298	4%
15	Fiber recovery cleaner rejects	1.181	1.074	1.090	1.094	1.043	1.113	1.099	0.0464	4%
26	Dilution water to fiber recovery	-	-	-	-	-	-			
11	1st thru-flow cleaner feed	0.457	0.499	0.482	0.491	0.493	0.480	0.484	0.0149	3%
16	1st thru-flow cleaner accepts	0.523	0.571	0.550	0.561	0.533	0.540	0.546	0.0179	3%
17	1st thru-flow cleaner rejects	0.100	0.088	0.101	0.105	0.119	0.112	0.104	0.0106	10%
16	2nd thru-flow cleaner feed	0.523	0.571	0.550	0.561	0.533	0.540	0.546	0.0179	3%
18	2nd thru-flow cleaner accepts	0.575	0.592	0.589	0.607	0.605	0.570	0.590	0.0150	3%
10	2nd thru-flow cleaner rejects	0.117	0.118	0.132	0.120	0.129	0.135	0.125	0.0077	6%
18	Lamort flotation cell feed	0.575	0.592	0.589	0.607	0.605	0.570	0.590	0.0150	3%
20	Lamort flotation cell accepts	0.546	0.577	0.555	0.596	0.608	0.630	0.585	0.0320	5%
21	Lamort flotation cell rejects	0.491	0.404	0.870	0.715	0.667	-	0.629	0.1848	29%
22	Pulp from drum washer	23.36	23.55	23.49	24.36	23.19	22.35	23.38	0.6479	3%
23	Washwater from drum washer	0.032	0.026	0.030	0.020	0.022	0.022	0.025	0.0050	20%

Note consistencies are all expressed as percent oven-dried solids.
SD = Standard deviation
COV = Coefficient of variation = 100% x SD/Ave.

APPENDIX 1: USPS LABORATORY RECYCLABILITY PROTOCOL

These procedures are developed for a laboratory recyclability process for environmentally benign postage stamp materials. The recyclability process consists of paper stock preparation, repulping, Valley flat screening, hand sheet making and analysis of con- taminants at various stages of the process. A flow diagram of the process is attached (Attachment 1). These procedures are designed to be consistent with the USPS Pilot Scale Recycling Protocol. This protocol is a living document and further modifications will be incorporated before the final protocol is issued. It is anticipated that these modifications will reflect minor changes.

A. EQUIPMENT & MATERIALS

I. Equipment

1. Balance capable of weighing to 0.1 gram with at least 3000 gram capacity
2. Graduated beaker of 3000 ml capacity, plastic
3. Pyrometer capable of measuring from 0 to 100°C (32 to 212°F)
4. pH meter
5. 15 L plastic bucket with pouring spout
6. 40 L graduated plastic tanks fitted with ball valve (2)
7. Heavy duty variable speed motor
8. 13 cm bow tie agitators (2)
9. Valley Flat Screen with a six cut (0.006" = 0.15 mm) chrome plated slotted screen and a 12 cut (0.012" = 0.30 mm) brass slotted screen; 4, 8 and 10 cut screens are optional.
10. Brass sieve, 200 mesh (75 micron openings), 12" diameter (30 cm), 8" high (20 cm) (2), and meeting ASTM E-11 specification.
11. While plastic spatulas, assorted sizes
12. Aluminum weighing pans
13. Plastic beakers, 600 ml capacity
14. 20 L graduated plastic tank fitted with ball valve
15. Variable speed motor
16. Graduated cylinder, 500 ml capacity, plastic
17. TAPPI or British sheet mold, circular, 6" (15 cm)
18. Couch plate and roll
19. Drying rings (70)
20. Drying plates (70)
21. Image Analysis system, such as Optomax or Apogee
22. Scanner, such as HP ScanJet 3C, 600 dpi, or equivalent
23. Image analysis standards
24. Analytical balance capable of weighing to 0.0001 grams
25. Polished chrome plated plates
26. Press, unheated, with 0 to 8 bar gauge, 20 cm x 20 cm platens
27. Process log Sheets (Attachments 2A and 2B)
28. Hose and nozzle
29. 0.5 cm shredder
30. Gallon, quart and pint glass jars
31. Oven, forced air, capable of maintaining 70° and 110°C (158 and 230°F)
32. Adirondack 450H laboratory pulper with high consistency rotor and auxiliary heater, or equivalent.
33. Camera
34. Denver D-12 laboratory flotation machine.
35. Hot plate with stirrer
36. 1000 ml volumetric flask

II. Materials

1. White copy paper, 20# weight, with a contaminant level, as measured by USPS Image Analysis Protocol Pre-Stained Procedure, of no greater than 5 PPM.
2. White wove envelope paper, 24# weight, with a contaminant level, as measured by USPS Image Analysis Protocol Pre-Stained Procedure, of no greater than 5 PPM.
3. PSA stamp or laminate
4. Morplas Blue 1003, 0.1 by weight in heptane, filtered through Whatman #4 paper

5. Morplas Blue 1003, 0.1% by weight in methanol, filtered through Whatman #4 paper
6. Keystone Blue (Solvent Blue 58), 2% by weight volume in an 80/20 volume blend of isopropanol/tolulene.
7. Methanol
8. Blotters, 8" square, meeting specifications described in TAPPI T 205
9. Whatman #4 filter paper
10. Water supply, filtered, control heated up to 60°C (140°F)
11. 1 N NaOH
12. 0.5% aqueous solution of High Point DI 700 or Vining Industries L787 or L779 flotation surfactant, or their equivalent
13. 1 N HCl

B. PAPER FEED STOCK PREPARATION

I. Quality of Materials

Face paper shall be both printed and unprinted.
Release liner will be both printed and unprinted.
Adhesive will be both stained and unstained.

II. Sample Configuration

Post-Consumer Feed Stocks

Sample A: 5% stamp (face paper/adhesive) /47.5% copy paper / 47.5% wove paper
Sample B: 1.5% stamp (face paper/adhesive) /49.25% copy paper / 49.25% wove paper (optional)

Pre-Consumer Feed Stock

Sample C: 10% laminate (fact paper/adhesive/release liner) /45% copy paper / 45% wove paper

III. Procedure

1. Weigh to the nearest 0.1 gram the proper amount of copy paper, wove envelope paper and sample. The total of copy paper, wove envelope paper and PSA material will be 360 grams oven dried (OD) for all tests.
   The amount of material to be used for all three samples is given in the table below:

Table 1: Materials To Be Used

Sample	Grams Stamp Material		Grams 1:1 copy: envelope paper blend	
	Bone Dry	CTH Cond.	Bone Dry	CTH Cond.[1]
A	18.0	18.9	342.0	359.4
B	5.4	5.67	354.6	372.7
C	36.0	37.7	324.0	340.5

For samples A and B, weigh a 19 cm x 19 cm sheet of unstained stamp laminate to 0.l g. (i) Remove the release liner and weigh the liner to 0.1g. (ii) Use the face stock paper plus adhesive for Step 6. Calculate the weight of bone dry stamp laminate needed to give the required weight from the formulae:

$$\text{Laminate Needed for A} = \frac{(18.0 \times (i))}{(i - ii)} \text{ grams}$$

$$\text{Laminate Needed for B} = \frac{(5.4 \times (i))}{(i - ii)} \text{ grams}$$

Record the data in the Pulping Notebook. This page will serve as the code number for labeling all samples.

[1]CTH = Constant Temperature & Humidity conditions of 23°C (77°F) and 50% relative humidity.

2. If the adhesive is NOT to be stained, proceed to Step 14.
3. Test a small portion of the adhesive to see that it is not visibly attacked by the dye solution. Choose the proper solvent for the dye.
4. Cut the laminate into 19 cm x 19 cm sheets prior to weighing.
5. Remove the release liner and set aside for future use.
6. Mount the face stock, adhesive side up, on two sheets of 20 cm x 20 cm blotter paper.
7. In a well vented fume hood, flood the adhesive with about l5ml of dye solution, by applying it evenly across the surface of the adhesive.
8. Swirl the dye solution so that all even color is developed as the solvent evaporates. Minimize the amount of dye going over the edges of the adhesive and staining the face paper.
9. Leave the dyed face stock in a hood until the solvent evaporates.
10. Continue air drying the face stock for thirty minutes.
11. Repeat steps 5 through 10 for the remaining 19 cm x 19 cm strips.
12. Bake the air dried strips for 5.0 minutes in a 70°C (158°F) oven.
13. Cool the strips and let them equilibrate to CTH conditions before laminating each of them to envelop paper weighed out in Step 1 for Samples A and B. For Sample C, laminate them to the release liner saved in Step 5. Laminate the adhesive with the 4.5 lb. roller. Condition the samples in the CTH room for a minimum of 24 hours but no greater than 72 hours before proceeding to Step 15.
14. For unstained samples, peel off the release liner and laminate the face stock to a sheet of wove paper for Samples A and B and condition them as described in Step 13.
15. Intersperse the laminated sample evenly between the remaining sheets of copy and wove paper.
16. Shred the sheets into 0.5 x 30 cm strips in a commercial paper shredder, making sure the collection basket is cleaned of all prior materials.

IV. Standard Conditions & Parameters

Pulping and screening

1. Temperature 46°C (115°F) ±1.5°C
2. pH: 10
3. Screening: Two Successive Screenings - Valley Flat Screen
   First Screen: 12 cut (0.012") [0.30 mm]
   Second Screen: 6 cut (0.006") [0.15 mm]

Flotation

1. Temperature 43°C (110°F)
2. Surfactant Level: 0.075%
3. Consistency: 1.0%

Hand Sheet Preparation

Before Screening:	15 hand sheets
After First Screening (12 cut):	15 hand sheets
After Second Screening (6 cut):	15 hand sheets
After Flotation:	15 hand sheets

C. PULPING

1. Add 2040 grams of filtered water, adjusted to pH 10 with 1 N NaOH, to the Adirondack pulper. Caution: Wear eye protection - NaOH is corrosive. Heat to 41°C (105°F)[2] with the auxiliary heater while stirring the water at 10 Hz. Measure the temperature with a pyrometer and note the chart recorder temperature. Caution: Use hand protection - pulper and water are hot.
2. Add the shredded paper feed stock to a 15 Liter plastic bucket. Add the pH 10 water from the pulper to it and with a plastic spatula manually wet out the shredded strips of paper. Caution: Use eye protection.
3. Carefully transfer all materials back to the pulper and loosen the wetted paper.

[2] Acceptable mill temperature range is 38-60°C (100-140°F).

4. Measure the temperature. If it is below 41°C (105°F), heat the batch until these temperatures are reached. Standardize the pH meter at 7.0 and 10.0.
5. Start the pulper at 150 RPM and start the timer and ramp it to its final speed of 600 RPM. The ramping process should be carried out as fast as possible without splashing any liquid out of the pulper.
6. Once the final speed is reached, continue pulping for a total of 8.0 minutes. If 600 RPM is too fast to give a good mixing and folding pattern, reduce the speed until such a performance is reached.
7. During the run, stop the pulper after 4.0 minutes to rinse down any unpulped material attached to the side of pulper; stop and restart the timer accordingly.
8. After pulping, reset the speed control to 10Hz and immediately measure the pH and temperature of the pulp at several places. Report the range of pH and temperature found. Optional: Take a picture of the high consistency pulp.
9. Dilute the batch with 4800 ml of 46°C (115°F) filtered water and mix at 150 RPM until the batch is homogeneous. Do not increase the speed too rapidly to avoid splashing the mix water out of the pulper.
10. Before emptying through the bottom ball valve into a 15 Liter bucket with a pouring spout, measure the pH and record the pulp characteristics using the code type described in Table 2. Take a photo of the pulp in the bucket.

Table 2: Characterization of Pulped Stock

Type	Description
1	Very large three-dimensional clumps of adhesive and several large clumps of adhesive. Adhesive is very tacky, tough and clastomeric.
2	A mixture of large round three-dimensional particles and smaller flat particles. Adhesive is tacky and elastomeric.
3	A mixture of few large round three-dimensional particles and large and small two-dimensional particles. Adhesive is not tacky or elastomeric.
4	A mixture of large and small two-dimensional particles, with more smaller particles than in Type 3 adhesives. Adhesive is not tacky or elastomeric.
5	Partially soluble/dispersible with bluish color in the aqueous phase. Contains blue floaters. Very large number of very small sperical particles.

I I Transfer the material in the bucket into the 40 liter Valley flat Screen feed tank. Caution: Use hand protection - material is hot.
12. Using the hose, rinse the pulper with 46°C (115°F) filtered water and fill the pulper about two thirds full. Mix at 10Hz for a few minutes, then dump the material into the 15 Liter bucket. Add this materal to the flat screen hold tank. Use hand protection as metal nozzle will get hot.
13. Repeat step 12 until all pulp fibers are transferred to the flat screen feed tank. Reverse the direction of the pulper for the last rinses until no more pulp is transferred.
14. With the bow tie agitator blade positioned nearly at the bottom of the feed tank, start adding 46°C (115°F) filtered water from the hose to the feed tank. When the tank is at the 20 Liter mark, start the agitator at 300 RPM. Caution: Use hand protection, as metal nozzle will get hot.
15. Continue adding water until the final volume of 40 Liters is reached. Just prior to this time, shut off the agitator to eliminate any vortex effects that would affect the accuracy of the volume measurement. The consistency of the batch will be about 1.0% at this time.
16. Continue mixing the batch for 15 minutes.
17. Clean the pulper, removing all traces of debris.
18. Remove 2500 ml of the batch, return it to the feed tank, then take a 3750 ml sample and transfer it to the Dolar feed tank for the hand sheet maker.
19. Add 7500 ml of cold water to the Dolar tank and mix for five minutes.

20. Turn on the laminating press used to dry hand sheets. Be sure the temperature is set for 100°C (212°F).
21. Make a hand sheet using 400 ml of the Dolar feed stock. Dry the sheet between two clean blotter sheets on each side and press out excess water at 3.7 bar. Replace the blotter papers with new ones and dry five minutes at 100°C (212°F). If needed, replace wet blotters with new ones and continue drying in the press until the hand sheet reaches constant weight. Label it on the outer 0.5 cm of the sheet by the last three digits of the notebook code followed by Bw.
22. Adjust the amount of pulp to be used for making a hand sheet according to the formula: 400 x 1.20/ wt. of sheet (g).
23. Make 15 "before screening" hand sheets according to Tappi T-205 proce- dures but with only a two minutes pressing. Air dry the hand sheets. Label these similarly with the three digit code and the consecutive sheet number starting with 1. Return unused pulp to the flat screen feed tank.
24. Clean the agitator, Dolar feed tank and graduated cylinder.

D. VALLEY FLAT SCREENING WITH 12 CUT SCREEN

1. Remove the 200 mesh (75 micron opening) brass screen from the accepts tank, and start flushing the Valley flat screen with 46°C (115°F) filtered water for a few minutes. Be sure that the drain ball valve of the accepts tank is fully open.
2. Shut off the water to the flat screen and let all the water drain from it. Remove any debris from the screen.
3. Replace the 200 mesh sieve and start the flow of 46°C (115°F) water to the flat screen. Adjust the out flow valve such that a 7-9 cm head is maintained in the flat screen box. Record the temperature of the water in the box.
4. Position the flat screen feed tank ball valve such that the feed stream will be as close as possible to the inlet water stream of the flat screen.
5. Start the flow of pulp after starting the flat screen motor. Adjust the flow rates of the pulp feed stream and the accepts outlet stream to maintain equilibrium conditions. Note the time, temperature and volume of the feed tank. The strip chart recorder may be used to monitor the temperature of the flat screening.
6. Periodically, as needed, use a plastic spatula to sweep the fiber accepts away from the discharge area to permit rapid flow of the waste water from the 200 mesh sieve. Transfer the dewatered pulp to a second vessel to renew the screen.
7. Record the temperature of the Valley flat screen at the 30, 20, and 10 Liter mark and near the end of the run. At the 20 Liter mark, also measure the temperature of the pulp in the feed tank.
8. Take a pilot of the flat screen at the 20 Liter mark and at any other appropriate times.
9. Reduce the speed of the agitator as the volume of the pulp decreases, to avoid uneven mixing and splashing of the pulp on the walls of the tank.
10. Rinse the feed tank with 46°C (115°F) water as needed to assure transfer of all the pulp to the flat screen box. Continue the flow of water until the liquid in the box becomes clear. Then continue rinsing using cold water to harden the stickies to make their recovery easier. This will take several minutes.
11. Shut off the water flow into the box, by first turning off the valve of the inlet pipe. Let all the water drain into the accepts tank. Take a photo of the Valley Flat Screen.
12. Use the plastic spatula to squeeze excess water from the fiber accepts.
13. Use the white plastic spatulas to transfer the rejects to a tared aluminum dish. At this time, do not be concerned with not obtaining a quantitative transfer of all the rejects.
14. Restart the Valley flat screen using cold inlet water. Use a spatula and wash bottle to remove contaminants stuck to the sides of the box. Shut off the inlet water and let all the water drain from the flat screen

IS. Use spatulas, and/or tweezers to remove all contaminants. Where convenient, separate fiber from adhesive and weigh fibers separately.

16. Repeat steps 14 and 15 until all contaminants are transferred to the tared aluminum dish. Set aside and take 2 single photo of it together with the 6 cut rejects.
17. Dry rejects at 105°C to constant weight. Save the dried material for further analysis.
18. Wash the feed tank thoroughly to remove all pulp particles. Remove the ball valve and remove any debris held up in the valve. Reassemble the valve to the tank.

19. Remove the 12 cut screen and transfer all material which passed through the screen into the accepts of Step 12. Clean the screen plate with water and a 5 mil plastic shim. If the plate is not clean and tack free, clean it with an appropriate solvent. Qualitatively record the case of cleaning the pulper, the feed tanks, the Valley Flat Screen and the slotted screens using the code in Table 3.

Table 3: Characterization of the Ease of Cleaning

Classification	Description
Very Easy	Cleans up with only a water stream and sponge
Easy	Cleans up with a water stream, sponge and brush, and occasional use of plastic shim
Normal	Cleans up with a water stream, sponge and brush, and use of plastic shim
Difficult	Requires a water stream, sponging and brushing,and extensive use of a shim to remove adhesive
Very Difficult	Requires solvent cleaning of any equipment

20. Install the six cut screen plate in the Valley Flat Screen.

E. HAND SHEET PREPARATION WITH 12 CUT SCREEN ACCEPTS

1. To a separate clean flat screen tank, and all the fiber accepts and fill the tank to the 10 gallon mark with 46°C (115°F) filtered water. Start the bow tie agitator when the tank is about half full at 300 RPM.
2. When the tank is full, continue agitation for at least 10 minutes at a RPM that induces a small vortex.
3. While the pulp is being gently agitated, transfer 10 Liters of the mix to the Dolar tank.
4. Fill the Dolar tank to the 20 Liter mark with cold filtered water, and begin agitation with the bow tic agitator at a 70 motor setting. Mix for five minutes.
5. Turn on the beater to the laminating press and set for 100°C (212°F).
6. Make two hand sheets using 400 ml of the mix according to Tappi T 205 procedures. Dry the sheets in the press to constant weight, and based on the average weight of the hand sheets, calculate the amount of pulp needed for 1.2 g hand sheets, as described in Step 23 of Section C. Label the sheet with the digit code number followed by - 12w. If the volume of pulp lies outside the range of 350 to 450 ml, dilute with cold filtered water or add pulp from fiber accepts tank as needed.
7. Make at least 15 “after 12 cut flat screening” hand sheets. Mark each in the order made and with a product code. The product code will be the last three numbers of the notebook page used to record the Pulping data. The order will start with the number “16”.
8. Air dry the hand sheets overnight under ambient conditions.
9. Continue air drying the sheets under CTH conditions, 73°F, 50% RH, for 24 hours.
10. Carefully remove the hand sheets from the plates to prevent curl or wrinkling and weigh the hand sheets and calculate the average weight.
11. Store the hand sheets in a sealable plastic bag, labeled with the date, Pulping notebook page number, and five digit product code.
12. Return unused pulp in the Dolar tank to the Valley Feed Tank.

F. VALLEY FLAT SCREENING WITH 6 CUT SCREEN

Repeat Steps 1-19 of Section D.

G. HAND SHEET PREPARATION WITH 6 CUT SCREEN ACCEPTS

1. To the clean flat screen tank, add all the fiber accepts and fill the tank to the 24 Liter mark with 46°C (115°F) filtered water. Start the bow tie agitator when the tank is about half full at 300 RPM.
2. When the tank is full, continue agitation for at least 10 minutes at a RPM that induces a small vortex.
3. While the pulp is being gently agitated, transfer 6 Liters of the mix to the Dolar tank.

4. Fill the Dolar tank to the 20 Liter mark with cold filtered water, and begin agitation with the bow tie agitator at a 70 motor setting. Mix for five minutes.
5. Turn on the heater to the laminating press and set for 100°C (212°F).
6. Make two hand sheets using 400 ml of the mix according to Tappi T 205 procedures. 7. Dry the sheets in the press to constant weight, and based on the average weight of the hand sheets, calculate the amount of pulp needed for 1.2 g hand sheets, as described in Step 23 of Section C. Label the sheet with the digit code number followed by -6w. If the volume of pulp lies outside the range of 350 to 450 ml, dilute with cold filtered water or add pulp from fiber accepts tank as needed.
7. Make at least 15 "after 6 cut flat screening" hand sheets. Mark each in the order made and with a product code. The product code will he the last three numbers of the notebook page used to record the Pulping data. The order will start with the number "31".
8. Air dry the hand sheets overnight under ambient conditions.
9. Continue air drying the sheets under CTH conditions, 73°F, 50% RH, for 24 hours.
10. Carefully remove the hand sheets from the plates to prevent curl or wrinkling and weigh the hand sheets and calculate the average weight.
11. Store the hand sheets in a sealable plastic bag, labeled with the date, Pulping notebook page number, and five digit product code.

H. FLOTATION

1. Preparation of 0.5% HP DI 700 Solution
    a. Prepare 0.5% High Point DI 700 solution by weighing out 5.00 grams of 100% HP DI 700 in a 500 ml beaker.
    b. Add ~200 ml heated (~ 105°F, 40°C) distilled water into the beaker.
    c. Stir the solution until the D.I 700 dissolves in the water.
    d. Transfer the solution into a 1000 ml volumetric flask.
    e. Rinse the beaker three times with a small amount of distilled water and transfer to the flask.
    f. Add distilled water to the neck mark of the flask.
    g. Shake the flask and mix the solution well.
    h. Pour the solution into a container labeled date, and concentration of solution
2. Close the air valve at the top of the standpipe of flotation machine.
3. Transfer 9.0 liters of 1% consistency pulp in the 6 cut accepts feed tank into a clean 10 liter flotation cell.
4. Lower the float assembly to the lowest level and then back up one level.
5. Adjust the temperature of pulp to 110°F (43°C) using a heating core.
6. Transfer 13.5 ml HP DI 700 at 0.5% concentration into a small beaker.
7. Pour the DI 700 into the flotation cell and rinse the beaker with a small amount of water twice and add the water to the cell.
8. Turn on the switch to mix the pulp and surfactant for five minutes.
9. Turn on the air valve gradually to full open and at the same time start the timer and skimming device, running at 18 RPM by using the high speed range of the motor controller at an 8 setting.
10. Take a photo after 1 minute and 4 minutes.
11. Turn off the flotation cell and the skimming device after 5.0 minutes.
12. Move away the skimming device and raise the float assembly to its uppermost position.
13. Remove the container with flotation rejects.
14. Remove the contaminates (inks, stickies, etc.) left on the wall of the cell using a spatula carefully and transfer to the container with flotation rejects.
15. Filter the flotation rejects with a Büchner funnel on a weighed filter paper (#4 Whatman Filter Paper) and take a picture for the rejects.
16. Transfer the flotation pulp accepts into a five gallon tank.
17. Repeat the Steps 2-16.
18. Mix well the flotation accepts from the two runs in the 20 Liter tank.
19. Transfer six liters of pulp from the 20 Liter tank into another 20 Liter tank and fill it with cold water.

20. Make 15 hand sheets using the pulp in 20 Liter tank.
21. Dry the flotation rejects to constant weight in a 105°C oven.
22. Calculate the weight percentage of flotation rejects.

I. CLEAN UP PROCEDURES

1. Remove the flat screen using the brass jack screws and clean the surface and slots with a sponge and water.
2. Clean the sides of the Valley Flat Screen and the plastic feed tanks with water and a sponge, and record case of cleaning.
3. Power wash the screen to remove last traces of contaminants. Visually examine the slits for any hold up. Remove traces of fiber and/or adhesive with the appropriate size plastic skim. Never use a metal shim, as it will increase the slot width.
4. If needed, clean the plate with the appropriate solvent in a well vented hood.
5. Clean out any debris that formed under the vibrating flat screen before replacing it.
6. Repeat procedures B through G using only clean copy paper, 360 grams, for the paper feed stock. Forced dried hand sheets can be used for image analysis. Only ten hand sheets have to be made from the flat screen accepts. Process temperatures of 40-50°C can be used, and pulping carried out at neutral pH, using only the 6 cut screen.
7. To determine if acceptable contaminant levels are reached, repent Steps 1 and 2 of Section K. If unacceptable levels are found, repeat Steps 1 through 6 of Section I until the acceptable level is reached. For both pre- and post-stained feed stocks, use the appropriate image analysis conditions described in the USPS Image Analysis Protocol. If the PPM is greater than 5 and the CT/M^2 is greater than 145, consider running another clean out batch.

J. VALLEY FLAT SCREEN REJECTS ANALYSIS

1. Describe the physical form of the dried rejects and give an estimate of the amount of fiber it contains.
2. Calculate the percent rejects based on the dry weight of the adhesive in the rejects obtained in Step 17 of Section D and the original weight of the adhesive.

K. HAND SHEETS ACCEPTS ANALYSIS

1. Perform image analysis measurements on the hand sheets according to the USPS Image Analysis Protocol appropriate for either pre-stained or post-stained hand sheets.
2. Report the mean values for PPM, $Count/m^2$, spec size distribution for the "before screening". "after each screening" and "after flotation" hand sheets only. Measure brightness by TAPPI T-442 on the unstained “after flotation” hand sheets. Report the percent reduction in mean PPM for each unit operation. For printed feedstocks, separate the ink particle contaminates from adhesive contaminates in printed feed stocks by subtracting the PPM and CT/M^2 obtained from the unstained hand sheets from the corresponding values of hand sheets that have been stained and washed by the USPS Image Analysis protocol.

L. REPORTING

1. The report shall consist of the following:
   a. Copies of Data Log Sheets (see Attachment 2).
   b. Weight of rejects from both the 6 and 12 cut screens and flotation.
   c. Image analysis data including equipment settings, number of hand sheets analyzed, mean values for PPM, counts per square meter, PPM standard deviation, distribution of sizes of specs, and efficiency of each operation.
   d. Qualitative description of clean-up performance, using the code in Table 3.
   e. A mass balance analysis for the adhesive.
   f. Contaminant levels for both the adhesive and ink particles when applicable.
   g. An overall assessment of the recyclability, using the criteria that PPM after flotation should meet the target of 20 PPM for post-consumer feed stocks and 35 PPM for pre-consumer feeds. Both feeds should give acceptable clean up performance.

Attachment 2A: Recyclability Log Sheet

1	Date:
2	Laminate No:
3	Notebook No:
4	Sample Configuration:
5	Adhesive Dye Solvent:
6	Wt. Envelopes (g): Face: Liner:
7	Wt. Carrier Paper (g):
8	Wt. Pulper Water (g): Temp Start: pH:
9	Pulping Start Time:
10	Total Energy Consumed:
11	End Temp:
12	pH @ 5%
13	Pulp Type:
14	Before Hand Sheet Volume (ml): Dried Weight (g): Volume Used: Number Made: Press (1 or 2): Avg. weight (g):
15	12 Cut Valley Flat Screen Start Time: Volume Time Temp. 40 L 30 L 20 L 10 L 4 L Pulp Addition: Total: Rejects Weight (g): Description: Adhesive (g):
16	12 Cut VFS Hand Sheets Volume (ml): Dried Weight (g): Volume Used: Number Made: Press (1 or 2): Avg. Weight (g):
17	6 Cut Valley Flat Screen Start Time: Volume Time Temp. 40 L 30 L 20 L 10 L 4 L Pulp Addition: Add'l Time: Total: Rejects Weight (g): Description: Adhesive (g):
18	6 Cut VFS Hand Sheets Volume (ml): Dried Weight (g): Volume Used (ml): Number Made: Press (1 or 2): Avg. Weight (g):
19	Comments

Attachment 2B: Flotation Log Sheet

Date:		
Sample No:		
Notebook No:		
Pulping Configuration:		
	FLOTATION A	FLOTATION B
Pulp Weight (g):		
Pulp Consistency (%):		
Start Temperature (°C):		
End Temperature (°C):		
HP DI 700 @ 0.5% Concentration (ml):		
Mixing Time (min.):		
Flotation Time (min.):		
Flotation Cell Motor Speed (RPM):		
Air Valve Setting:		
Skimming Device Settings:		
Total Reject Weight (g):		
Reject Weight Based on OD Pulp (%):		
Test Volume for Each Hand Sheet:		
Volume (ml):		
Dried Weight (g):		
Volume Used (ml) / Hand Sheet (1.2 g):		
Number of Hand Sheets Made:		
Average Air Dried Weight (g):		
Comments:		

APPENDIX 2: Pilot Laboratory Recycling Procedure

These procedures are developed for evaluating the recyclability of environmentally benign postage stamp materials using those conventional unit operations located in the USDA Forest Products Laboratory pilot laboratory paper recycling facility. The process involves high consistency repulping, slot screening, centricleaning with both forward and flow-through cleaners, flotation, washing, and papermaking using a pilot laboratory machine. Screening and forward cleaning include secondary fiber recovery.

Alternate facilities used to evaluate experimental adhesives/constructions should mimic as closely as possible the sequence of unit operations and operating conditions employed here. It is recognized that there will be equipment variations from plant to plant.

A. Equipment & Materials

I. Equipment

1. VOITH high consistency pulper model HC-1.5, 2500 L total volume, 1500 L maximum working volume, equipped with OHIO SEMITRONICS model CTX-500S current transducers feeding an OHIO SEMITRONICS model WH3-11195 AC watt-hr meter linked to a personal computer to provide energy consumption data.

2. Voith MULTIFRACTOR pressure screen model 00 equipped with a 0.30 mm (0.012") slotted basket (1 st primary stage). Voith VPS-05 pressure screen equipped with a 0.10mm (0.004") C-bar slotted basket (2nd primary stage).

3. AMINCO 6-plate vat flatscreen w/0.2 mm (0.008") slotted plates 600-liter tank plus transfer pump (fiber recovery and adhesive collection stage).

4. Beloit POSIFLOW 60 forward centricleaner (1st primary stage); Celleco CLEANPAC 70 forward centricleaner (2nd primary stage); Voith KSP 60 forward centricleaner with primary reject collection tank and stock pump (secondary fiber recovery stage).

5. Beloit UNIFLOW HF through-flow centricleaner, 100 mm (4") nominal diameter; two primary cleaners in series.

6. LaMort 5000-L, 2-stage laboratory flotation cell.

7. Scales and 200-Liter drums to collect secondary forward cleaner rejects and flotation cell rejects.

8. Stock tanks and pumps, sufficient to store and transfer stock being processed

9. Foxboro 8000A Magnetic flow tubes and IMT25 Magnetic flow transmitters installed throughout the system. These are the primary units for rate and volume measurements

10. NALGENE, HDPE containers, or equivalent: 1-L, 4-L, 10-L, and 20-L capacities in sufficient numbers for sample collection.

11. Self-sealing plastic bags, for sample collection: 4-L plus others as needed

12. Assorted plastic bags (30 x 60 cm, 45 x 90 cm minimum sizes) for sample storage. Scales, 150-kg minimum capacity, minimum resolution 0.1 kg. Thermometers, utility knives, glassware, pH meter, etc. as required.

13. Screen carts, 1 x 1 x 1.5 meter, with 100- mesh bottom wire (6 needed)

14. Balance, 1-kg capacity, minimum accuracy 0.1 gm.

15. Vacuum drum washer, laboratory unit with 100-mesh wire; equipped with press roll and shredder.

16. Barrels, 200-Liter (or equivalent) for storage of cleaned pulp.

17. Experimental paper machine and auxiliary equipment

18. TAPPI handsheet former and auxiliary equipment

19. OPTIMAX Speckcheck Analyzer with a HP scanner

II. Materials

Definitions

Standard (Linered) Pressure Sensitive Adhesive (PSA) Stamp Laminate: Face Papers/PSA/Release Liner, with or without printing.

Face paper: base paper sheet only, as used to prepare the Standard (Linered) Pressure Sensitive Adhesive Stamp Stock described above.

Linerless Pressure Sensitive Adhesive (PSA) Stamp Stock: Release coating/ Printing/Face Paper/PSA.

Papers

Copy paper: single supply for all work; procured by USPS/FPL as a 31,000 lb. shipment derived from 2 consecutive master rolls from a commercial paper machine, slit into 24" wide working rolls of approximately 1100 LB each.

Envelope paper: single supply for all work; procured by USPS/FPL as a 180,000 LB shipment derived from consecutive master rolls from a commercial paper machine, slit into 24" wide working rolls of approximately 1100 lbs. each.

Stamp stock face papers: 45 kg. (approximate) supplied by USPS with tie layer, with no printing or adhesive. 3 rolls of paper, one roll from each of the suppliers.

Linerless (PSA) stamp stock: 50 kg. supplied by USPS. Same stock sample used for Phase 3 study.

Standard PSA stamp laminate: 150 kg. supplied in 3 rolls: conventional construction currently used for stamps (same supply used for phase 3 study)

Experimental PSA stamp laminate, as submitted to FPL through USPS/STR: 20 kg each adhesive on each face paper (3 preconsumer samples per adhesive) (60 kg total), procured as defect-free production scrap from the stamp preparation process.

Gummed WAG (water-activated glue) stamp stock; 3 rolls each printed by a different printing method: intaglio, offset & rotogravure.

PSA linerless label stock (color: "canary"), 80# roll. Current type used by USPS for change-of-address labels

USPS self-sticking PSA Label 11-F (July 1997): ***EXPRESS MAIL*** address label. 4000 labels (32 kg.).

III. Other materials

1. Morplas Blue 1003, 0.67 gm/L in heptane.

2. pH standards: 4.0, 7.0, 10.0

3. Blotters, 20 cm square, meeting TAPPI specifications

4. a) Whatman #4 filter paper or equivalent, 15 cm diameter.
   b) Same, 32 cm diameter.

5. 10-15% NaOH solution.

6. 10-15% H_2SO_4 solution.

7. Flotation aid: HighPoint DI-700A

8. Soft water: 20 and 80°C, central supply.

B. Stock Preparation (for each trial)

Throughout the following unit operations flowrates, consistencies, volumes, etc. will be determined in order to establish a mass balance for the processing. Necessary data will be recorded as appropriate.

I. Pulping

1. Weigh out 112.5 kg, oven-dry basis, of fiber-based material. Measure and record solids content of paper(s).

   A. A typical phase 4 pre-consumer loading will be 50.625 kg copy paper (45% of total), 50.625 kg envelope paper (45% of total) and 11.25 kg total composite stamp stock (10% of total).

   B. A typical phase 4 post-consumer loading will be 112.5 kg of cancelled envelopes each containing one sheet of copy paper,

   C. For a post-consumer, non-envelope, trial (phase 3 technique) the 11.25 kg of total composite stamp stock is weighed out, the release liner is removed and the remaining "stamps" are affixed to envelope paper. The removed release liner is weighed and an equivalent amount of copy/envelope paper (1/2 each) is added as make-up. This technique will approximate a 47.5% copy paper, 47.5% envelope paper, 5% stamp stock composition: the actual composition will depend on the basis weight of the release liner.

2. Fill pulper with 780liters softwater at 46±2°C (115±5°F). Adjust pH to 10.0 with NaOH. Use H_2SO_4 to correct overshoots.

   Pre-consumer trials

   Add copy and envelope papers, with minimal agitation until all paper has been introduced and wetted. Blend for 1 minute, adding only enough water to cause slurry to roll properly. Record total water added.

   Stop agitation at end of the one minute; measure pH and temperature. Restart pulper and immediately start energy-measuring unit. Blend in adhesive-containing stock quickly, while continuing to pulp for a total of 20 minutes. Add just enough water to maintain rolling of the slurry. Collect a consistency sample when rotor has stopped immediately upon shutting off pulper.

   Post-consumer trials

   Same as (a) except add cancelled envelopes in their entirety as quickly as pulper will take them. Add water only as necessary. Measure temperature and pH. Then start pulper and energy measuring unit.

(Target: total volume = 900 liters to give a 12.5% consistency. Actual volume and consistency is determined by producing the proper roll of the slurry at the highest possible consistency (minimum amount of water) to achieve the roll.).

(*Note: pulping time was determined in developing the phase 3 portion of this study.*)

3. At end of 20 minutes first shut off energy-measuring unit, then immediately the pulper, Measure final temperature, and pH, and record energy input. Dilute slurry to 1500 L, maintaining temperature at 46±2°C (115±5°F).

4. With minimal agitation, transfer slurry to screening feedtank via bull screen pit and sump pit, adding post-pulper 46°C dilution water only as necessary to effect transfer. In the feedtank dilute slurry to **2.265%** consistency maintaining temperature at 46±2°C, (115±5°F). Mix well. Measure actual consistency. Measure temperature. From stock in tank save one 10-L sample **(SAMPLE POINT 1).**

II. Pressure Screening

1. Pre-fill flatscreen, equipped with 0.2 mm (0.008") slots, and its 600-L stock tank with 46°C water. Record volume of water used. Fill pressure screens with water. Begin pumping flatscreen accepts weir water to 1st pressure screen, equipped with 0.30 mm (0.012") slots, at **175 L/min.** Adjust rejects flow to **45 L/m** (15% of total feed) with rejects passing back to flatscreen.

2. Begin pumping feedtank slurry to 0.30 mm pressure screen at **125 L/min** target rate total flow = 300 L/min), with accepts passing at **255 L/min** to the second pressure screen, equipped with 0.10 mm (0.004") slots. Pass 1000 liters of the initially watery 0.1 mm accepts from 2nd screen to the 600-liter flatscreen stock tank at **200-205 L/min** Rejects from the 0.1 mm pressure screen pass back to the feedtank at **50 L/min**

3. Pump the initially clear water from the 600-L stock tank to the flatscreen at **130 L/min.** When the 0.1 mm pressure screen accepts volume reaches 1000 liters divert these accepts to the 4000/8000-L stock tanks. Immediately begin adding sufficient fresh 46°C soft water to 600-L stock tank so as to maintain the 130 L/min flow to the flatscreen. Pressure screen target pressures are to be 115 kPa (17 psi) feed/ 110 kPa (16 psi) accepts for the 0.30 mm slots and 102 kPa (15 psi) feed/ 96 kPa (14 psi) accepts for the 0.10 mm slots. Actual values are determined by the nature of the stock and are to be logged and reported. Flowrates are to be maintained.

4. When at equilibrium save a 10-L carboy of **accept** stock from each pressure screen and a 20-L sample from the flatscreen accepts stock for handsheets (**SAMPLE POINTS 3, 5 & 7**). Measure consistency of all accepts and (**SAMPLE POINTS 2 & 4**) streams. Measure flowrates and total flows of all streams. Measure temperature of final accepts.

5. When 0.1 mm screening accepts reaches 10,500 liters (9,500 liters in accepts tank) shut off all flows. Record all volumes. Then pass all remaining flatscreen accepts back to feedtank for disposal. Flush flatscreen with fresh water to remove all fiber.

6. When only adhesive and paper tags remain on flatscreen collect rejected adhesive retained by the flatscreen, dry, and weigh for yield (**SAMPLE POINT 6**).

7. Dilute (at 46°C) accepts to 0.65% consistency, intermixing the slurry in the 4000- and 8000-L tanks until of uniform consistency. Save a 10-L carboy of the uniformly blended accepts slurry for handsheets (**SAMPLE POINT 8**). Measure consistency.

All of the next operations are performed as a continuous operation

III. Forward Cleaning

1. Slowly open feed valve to 1st primary forward cleaner (Beloit Posiflow 60) until accepts flow is 234 L/min. Feedrate will be between 240 and 260 L/min. Accepts flow to a 600-L stock tank and then are pumped to the 2nd primary forward cleaner (Celleco Cleanpac 270) at a feedrate that maintains the level in the 600-L tank.

2. Accepts at 212 L/min pass to a second 600-L stock tank. Back pressure of Cellecocleaner must be in the range of 54-136 kPa (8-20 psi). When flow is at equilibrium save a 10-L accepts sample from each cleaner **accepts** tank (**SAMPLE POINTS 9 & 11**). Rejects from both primary stages (56 L/min) are to be combined in a stock tank, diluted with fresh water (30 L/min) at 46°C, pumped through a secondary fiber recovery forward cleaner (Voith KS60P), with the accepts passing back to the primary forward cleaner feed tank and with the rejects (7-8 L/min) being collected in 55-gallon drums (**SAMPLE POINT 15**). Actual feed/accepts pressures to be set to balance flow. Measure consistency of rejects from each cleaner (**SAMPLE POINTS 10, 12 & 15**). Save two 10-L samples of secondary cleaner feedstock (**SAMPLE POINT 13**).

   Actual diluted consistency of primary rejects (***SAMPLE POINT 13***) *to be such that the secondary accepts are equal to the initial primary feed consistency.*

3. Save a 10-L sample of the secondary forward cleaner accepts for handsheets (**SAMPLE POINT 14**). Measure flow rates on all primary and secondary accepts and reject streams. Measure consistencies on all streams. Measure accepts temperature in both 600-L tanks and that of the secondary cleaner accepts.

4. As the 8000-L feedtank empties, continuously transfer contents of 4000-L feedtank to the 8000-L tank. When 8000-L tank is 1/2 empty draw down the 1st 600-L accepts tank by increasing the flow rate to 2nd primary cleaner to about 250 L/min Objective is to have this tank very nearly empty when main feedtank is empty. Similarly, if necessary, increase the feedrate to the secondary fiber recovery cleaner to draw down the primary rejects dilution tank.

IV. Through-flow Cleaning

1. When the 600-L 2nd forward cleaner accepts tank is 1/2 full of stock begin feeding slurry to the 1st through-flow cleaner at 212 L/min, adjusted slightly to maintain level in the feed tank. Target feed pressure is to be 210 kPa (30 psi) and hack pressure is to be 70 kPa (10 psi). Rejects are fed to a drain at approximately 22 L/min while measuring the reject flowrate and total reject volume with an accumulator. Save a 4-L reject sample for consistency (**SAMPLE POINT 17**).

2. Accepts flow to a 1000-L stock tank at approximately 190 L/min When this accepts tank is about 1/2 full turn on pump to feed 2nd through-flow cleaner (same pressures), with accepts from 2nd stage flowing to its stock accepts tank at approximately 165 L/min Balance flows to maintain tank levels. Measure reject flow as for 1st stage, saving a 4-L reject sample for consistency (**SAMPLE POINT 19**).
3. While maintaining a pressure difference of 140 kPa (20 psi) adjust actual pressures to balance flows. When at system equilibrium save a 10-liter sample of each **accepts** stream (**SAMPLE POINTS 16 & 18**). Measure accepts stream temperatures and flowrates.

4. At the same time that the 2nd through-flow cleaner accepts begin flowing to its accepts tank begin injecting DI-700A flotation aid (0.10% dry fiber basis) to suction port of the transfer

pump. When 2nd stage accepts tank is about 1/2 full begin pumping stock to LaMort flotation cell. Transfer rate will be about 165L/min.

V. Flotation and Washing

When slurry begins to enter flotation cell turn on airflow to 4 L/min. When outer cell is filled to above return stock injection ports turn on main circulation/transfer pump with stock exit valve shut. When outer cell is nearly filled begin the vacuum removal of foam. When outer cell is full open exit valve sending accepts to the glass-lined tank.. Adjust exit valve to balance outflow to inflow. Rejects are to be collected in 55-gallon drums, weighed and their consistency determined (**SAMPLE POINT 21**). Reject rate is about 2 L/min. When glass-lined tank has received about 1/2 of the total slurry save a 10-L sample of the flotation cell accepts (**SAMPLE POINT 20**). Determine its consistency.

2. Begin pumping stock from glass-lined tank to drum washer at 115-125 L/min as soon as pump is flooded. Dewater stock using vacuum plus pressure roll, shredding the mat and depositing the resulting crumbed pulp in barrels. Calculate total washer wastewater volume from input volume minus barreled pulp volume. Collect a 4-liter washwater samples after about a 20-minute running time; determine consistencies on these samples (**SAMPLE POINT 23**).

3. When input to the flotation cell from the through-flow cleaner ceases, shut off the outflow from the flotation cell and let the cell operate batch-style for 10 minutes to strip remaining flotables. Then reopen outflow valve and empty cell to glass-lined tank.

4. Sample final crumb pulp entering barrels by catching a handful of pulp at each 1/3 barrel level and place the three handfuls in a 12" x 12" plastic bag (**SAMPLE POINT 22**). Mix well and determine consistency on the combined sample. Weigh barrels and calculate total oven-dry pulp. Repeat for each barrel.

5. After consistencies are done, combine the pulps in each bag into one batch, mix well and submit for handsheets.

C. PAPERMAKING

1. To be done only on clean pulps; pulps with high residual adhesive are to be skipped. Two paper machine runs per adhesive, one pre-consumer/one post-consumer. The three pulps having a common adhesive but different printing inks are to be blended for one paper machine run, subject to the cleanliness limitation).

2. Reconstitute the shredded pulp to a 3% consistency and adjust its freeness if necessary. Alkaline size as appropriate.

3. Introduce the slurry to the FPL laboratory paper machine making a 20 LB/3 MSF printing/writing grade sheet.

4. Upon completion of the run slit the paper from the reel and save samples for analysis.

D. CLEANING

1. For stock preparation, after each trial the system is to be thoroughly flushed with water, followed if necessary, by a scrubbing with copy paper, envelope paper and/or virgin lap pulp. The system is again flushed with water and a second copy/ envelope paper scrubbing follows, with a pulp sample taken after shredding (**SAMPLE POINT 16**). This sample is processed in the PIRA Stickie Deposition Tester. If dirt count exceeds the equivalent to a FPL 15 ppm an additional scrubbing with copy/envelope paper is made. Target level is below 10 ppm FPL.

2. Baskets from the pressure screen are manually cleaned of the adhering adhesive, with the removed adhesive dried and weighed if excessive in quantity. The weight is included as part of the rejects for the respective stage. The baskets are then cleaned with a pressure washer plus a manual scrubbing with an organic solvent to remove all remaining traces of adhesive.

3. The plates from the flatscreen are cleaned with the pressure washer and solvent but no effort is made to measure the residual traces of adhesive as it represents only a small trace of the total adhesive removed from the screen previously. The entire flatscreen is pressure washed while the plates are out to maximize the cleanliness of the unit. Mineral spirits are used if necessary.

4. For the paper machine the clean copy paper stock from the final stock preparation system cleaning sequence is used to thoroughly clean the paper machine. Samples from the reel are checked for residual dirt. A dirt count over 50 ppm PIRA (15 ppn FPL) off the reel will indicate that an additional cleaning sequence is to be made. Fresh copy paper will be pulped and used for additional cleanings.

E. HANDSHEET MAKING

1. For all accept samples collected in each trial, handsheets are prepared according to TAPPI test method T-205 om-88. Condition is the same for 24 hours at 23° C, 50% relative humidity.

2. Details are presented in the companion protocol prepared by the FPL Paper Test Laboratory attached as a separate appendix.

3. For all paper machine paper samples cut sheets to 6” x 6” and condition as in 1.

F. DIRT COUNT ANALYSIS

1. Dye each handsheet and paper machine sample sheet with an appropriate dye solution.

2. Scan handsheet/sample (samples include paper machine samples) (both sides of each handsheet) for contaminants having an area greater than $0.02mm^2$. (Companion protocol covering this analysis contains full details).

G. FINAL SUBMISSIONS INCLUDING FINAL REPORT

1. Submit samples of handsheets, paper machine runs, and a final report of procedures and results to USPS.

APPENDIX 3: USPS IMAGE ANALYSIS PROTOCOL

These procedures were developed to evaluate the contaminant present in samples from USPS Pilot Scale Recycling Protocol and USPS Laboratory Recycling Protocol. The trials are for the recyclability of environmentally benign postage stamp materials.

A. POST STAINED HAND SHEETS (Using Avery Dennison Staining Technique)

I. Equipment & Materials

1. Scanner-based Image Analysis Dirt Counter System (such as Apogee, Optomax or equivalent) with a HP 4C 600 dpi color scanner, or equivalent
2. Weighted scanner block (20 cm x 20 cm)
3. Camera lens wipes
4. Glass cleaner
5. Solvent Blue 58 (Keystone Oil Blue ZVM), available from Keystone Pacific Division, Santa Fe Springs, CA
6. Toluene, industrial grade
7. Isopropanol, industrial grade
8. Methanol, industrial grade
9. Three 170 mm x 90 mm crystallizing dishes, Pyrex number 3140-170, or equivalent
10. Blotting paper, 20 cm x 20 cm, available from VWR Scientific #28303-100, or equivalent
11. Balance, weighing to the nearest 0.01 gm
12. Forceps, filter
13. Spatula, lab-spoon
14. Pyrex beaker, 100 ml
15. Rubber gloves

II. Image Analysis (Unstained Hand Sheets)

1. Randomly select 15 hand sheets from the set that is to be analyzed by image analysis. Note: 40 hand sheets are to be analyzed for the final product.
2. Turn on Scanner-based Image Analysis Dirt Counter System. Allow the machine to warm up for one hour.
3. Clean scanner glass with glass cleaner and paper towel. Wipe scanner glass with camera lens wipe to remove any particles or lint material.
4. Do twenty scans with the lid closed to warm up the scanner bulb.
5. Open lid. Place stained hand sheet (wire side down) on upper right hand corner of scanner bed. Place weighted scanner block carefully onto the hand sheet. (Care must be taken so that the hand sheet stays in position).
6. Scan hand sheet.
7. Remove hand sheet from scanner bed. Examine scanner glass for adhesive residue. If there is residue, clean glass with glass cleaner and then wipe with camera lens wipe. If no adhesive residue present, wipe scanner glass with camera lens wipe.
8. Place next hand sheet onto scanner bed, repeating steps 4-5 until 15 or 40 hand sheets are scanned.
9. Print scanner output.
10. Report Count (in one square meter), the Parts per Million, and Standard Deviation of Sheet PPM, and average Speck Area.

III. Stain Preparation

1. Add 2.0 g of Solvent Blue 58 dye to a mixture of 200 ml toluene and 800 ml isopropanol.
2. Stir until dye is completely dissolved. Solvent Blue 58 may be substituted with Solvent Blue 35 or a mixture of Solvent Blue 35 and Solvent Blue 58. Note: All mixing of chemicals should be done in a chemical fume hood while wearing appropriate safety gear.

IV. Staining the Hand Sheet

1. Pour 150 ml of the dye solution into a crystallizing dish. Immerse the hand sheet into the dye solution for 5-10 seconds.
2. Remove the hand sheet from the dye solution and remove the excess dye solution by pressing it between two sheets of blotting paper.
3. Dry the hand sheet at room temperature in a ventilation hood for at least three hours.

V. Washing the Hand Sheet

1. Pour 150 ml of methanol into each of the two crystallizing dishes. Immerse the dyed, dried hand sheet into the first dish and swirl for about 10 seconds.
2. Remove the hand sheet and blot the excess methanol.
3. Repeat the washing procedure in the second dish of methanol. After the second washing, the hand sheet should have a white background with blue PSA particles if PSA is present. If some dyes still remain in the background, repeat the methanol washing until the dye is removed.
4. Dry the hand sheet at room temperature until it is completely dried.

VI. Image Analysis (Stained Hand Sheets)

1. Randomly select 15 hand sheets from the set that is to be analyzed by image analysis. Note: 40 hand sheets are to be analyzed for the final product.
2. Turn on Scanner-based Image Analysis Dirt Counter System. Allow the machine to warm up for one hour.
3. Clean scanner glass with glass cleaner and paper towel. Wipe scanner glass with camera lens wipe to remove any particles or lint material.
4. Do twenty scans with the lid closed to warm up the scanner bulb.

Open lid. Place stained hand sheet (wire side down) on upper right hand corner of scanner bed. Place weighted scanner block carefully onto the hand sheet. (Care must be taken so that the hand sheet slays in position).

5. Scan hand sheet.
6. Remove hand sheet from scanner bed. Examine scanner glass for adhesive residue. If there is residue, clean glass with glass cleaner and then wipe with camera lens wipe. If no adhesive residue present, wipe scanner glass with camera lens wipe.
7. Place next hand sheet onto scanner bed, repeating steps 4-5 until 15 or 40 hand sheets are scanned.
8. Print scanner output.
9. Report Count (in one square meter), the Parts per Million, and Standard Deviation of Sheet PPM, and average Speck Area.

VII. Image Analysis Parameters

Turn on the Image Analysis System and set the test parameters for the instrument. The settings for the Apogee and Optomax Image Analysis Systems are as follows:

Settings for the Apogee Image Analysis System

Threshold Value	140
Resolution	600/inch
Scanner Mode	256 gs
Area Setting	15cm round
Scan Image	Normal
White Level	227
Black Level	0
Dirt Histogram	Upper limit =>5.000; Lower limit = 0.02-0.029; BHC=45

The total area scanned for the 15 cm diameter hand sheet is 0.013273 square meters.

Settings for the Optomax Speck Check Image Analysis System

Detection Level	140
Video Mode	Normal
Shade Compensation	Off
Scan Resolution	600

Display Resolution	x5
Window X Start	0.1
Window Y Start	0.1
Window X Size	6.0
Window Y Size	6.0
Type of Frame	Circle
Circle Frame Radius	2.8
Square Frame Width	8.2
Frame Height	10.6
Save Calibration	No
Size File in Use	02-5
Brightness Pre-Scan	Off

The total area scanned for the 15 cm diameter hand sheet is 0.015889 square meters.

B. PRE-STAINED ADHESIVE

I. Stain Preparation

1. Add 0.67 grams of Morplas Blue 1003 dye to 670 grams of solvent. Solvents that can be used are heptane or methanol.
2. Stir until the dye is completely dissolved.
3. Filter the solution through #4 Whatman paper, or its equivalent.

II. Staining the Adhesive

1. Test a small portion of the adhesive to see that it is not visibly attacked by the dye solution. Choose the proper solvent for the dye.
2. Cut the laminate into 19 cm x 19 cm sheets prior to weighing.
3. Remove the release liner and set aside for future use.
4. Mount the face stock, adhesive side up, on two sheets of 20 cm x 20 cm blotter paper.
5. In a well vented fume hood, flood the adhesive with about 15ml of dye solution, by applying it evenly across the surface of the adhesive.
6. Swirl the dye solution so that an even color is developed as the solvent evaporates. Minimize the amount of dye going over the edges of the adhesive and staining the face paper.
7. Leave the dyed face stock in a hood until the solvent evaporates.
8. Continue air drying the face stock for thirty minutes.
9. Repeat steps 3 through 8 for the remaining 19 cm x 19 cm strips.
10. Bake the air dried strips for 5.0 minutes in a 70°C (158°F) oven.
11. Cool the strips and let them equilibriate to 73°F/50% RH before laminating to envleope paper (post-consumer) or release liner (pre-consumer).

III. Image Analysis

1. Use the same parameters as in Section A, VI, above.
2. Clean the glass plate of the scanner bed by spraying Windex cleaner on a piece of cheese cloth and then wiping the scanning area with it. Never spray the glass directly with the cleaner. Wipe the glass surface with another piece of dry clean cheese cloth. Wipe scanner glass with camera lens wipe to remove any particles or lint material.
3. Warm up the bulb according to the needs of each instrument. The Apogee system's software automatically defines this period. For the Optomax, 20 scans are made for this purpose. Be sure to delete all scans prior to proceeding to Step 4.
4. Calibrate the instrument using the internal 70 dot pattern standards. See table below for Apogee and Optomax parameters:

IMAGE ANALYSIS CALIBRATION			
Internal Calibration Stds.		Apogee	Optomax
Black Dot	Count	70	70
	Area (mm)	68.1 ± 0.3	68.4 ± 0.3
Dark Blue Dot	Count	70	70
	Area (mm)	78.7 ± 0.3	79.2 ± 0.3
Light Blue Dot	Count	70	70
	Area (mm)	72.0 ± 0.3	72.2 ± 0.3

5. Place the hand sheet on the scanner bed with the smooth side down, using the sheet number as a locating point. The sheet should be placed in the upper right hand corner of the bed. This number should align with a mark at the top of the bed and the right hand side of the sheet should just touch the right side of the scanner bed. Place weighted scanner block carefully onto the hand sheet. (Care must be taken so that the hand sheet stays in position).
6. Scan all hand sheets and save the data to the appropriate spreadsheet file.
7. Remove hand sheet from scanner bed. Examine scanner glass for adhesive residue. If present, clean glass with glass cleaner and then wipe with camera lens wipe. If no adhesive residue present, wipe scanner glass with camera lens wipe.
8. Place next hand sheet onto scanner bed, repeating steps 5-7 until all hand sheets are scanned.
9. Print scanner output.
10. Report the average values for PPM, counts/m^2, spec size distribution and gray scale brightness (if applicable). Report the number of hand sheets tested for the "before screening" samples, both "after screening" samples, and the “after flotation” samples, along with the instrument parameters used. Calculate and report the efficiency of each screening step from the percent reduction in PPM.

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