

RCA LRP-2

Laboratory Testing Protocol For Paper Labels Coated With Recycling Compatible Pressure Sensitive Adhesives

1. Scope

- 1.1 This test protocol is for the testing and qualification of post consumer recycling compatible paper label products and pressure sensitive adhesives (PSA's) intended for use on paper to paper label products that are in conformance with Executive Order 13148 Section 702 "*Greening the Government Through Leadership in Environmental Management – Environmentally Benign Adhesives*".

2. Referenced Documents

- 2.1 Federal – Executive Order 13148, Section 702
- 2.2 USPS-P-1238F U.S. Postal Service Specification, Paper, Stamp, Pressure Sensitive Adhesive, Section 60 (LRP-7)
- 2.3 USPS-P-1238F U.S. Postal Service Specification, Paper, Stamp, Pressure Sensitive Adhesive, Appendix 1: Qualified Products List (QPL)
- 2.4 RCA Specification for recycling compatible adhesives and labels
- 2.5 RCA MRP, Mill Recyclability Protocol
- 2.6 RCA IAP, Determination of Adhesives in Paper Handsheets by Image Analysis
- 2.7 RCA standards are available on the TLMI web site, www.tlmi.com, Environmental Committee page.

3. Significance and Use

- 3.1 This protocol is intended to serve a dual purpose. It can be used to qualify an adhesive or a label product as recycling compatible. For example, an adhesive manufacturer may want to qualify an adhesive as recycling compatible. A converter may want to qualify a label product line as recycling compatible.
 - 3.1.1 To enable qualification of a complete paper label laminate as recycling compatible in conformance with Executive Order 13148 Section 702.
 - 3.1.2 To enable qualification of a pressure sensitive adhesive as recycling compatible in conformance with Executive Order 13148 Section 702.
- 3.2 Paper label laminate
 - 3.2.1 In this standard, a product line is defined as a group of labels with essentially the same type of adhesive and facestock construction. Within the product line, the actual products can differ in size, color, adhesive coating weight, facestock basis weight, and type of release liner. The adhesive coating weight of the test sample shall be greater than or equal to the average adhesive coating weight of the product line.
 - 3.2.2 For the qualification of the same adhesive on different facestocks, refer to Laminate Sample Preparation Method B.
 - 3.2.3 For qualification of a paper label laminate, the release liner is removed. Labels are applied to wove envelope paper. The feedstock for the test method consists of 4.5% labels, 47.75% copy paper and 47.75% wove envelope paper. The adhesive loading in this feedstock is about 1% PSA for a fully coated label.
 - 3.2.4 Laminates approved as meeting these requirements shall be approved only for the actual laminate construction and PSA coating weights submitted for testing. All other requirements for the PSA laminate shall be as specified in whatever performance specification applies to the end use labeling application for which the PSA laminate is intended.
- 3.3 Pressure sensitive adhesives
 - 3.3.1 For qualification of a pressure sensitive adhesive, the adhesive is coated on the three facestocks as specified in Table 2. These facestocks represent most of the label types found in commerce that are expected to enter the paper recycling stream. A composite sample is made from these laminates as described in Section 4.5 "PSA Laminate Sample Preparation Method B". If these adhesive and coated samples are prepared in the laboratory rather than in a production process, the laboratory versions must be representative of the finished commercial products. For example, coating packages are typically added to water-based adhesives to control foam, modify rheology and improve wet out in commercial coating processes. Laboratory prepared samples of an adhesive must contain all of these modifiers such that they are representative of their commercial counterparts.

- 3.3.2 To perform the test, the release liner is removed from the labels. The labels are then applied to wove envelope paper. The feedstock for the sample consists of 4.5% of the composite facestock laminates, 47.75% copy paper and 47.75% wove envelope paper.
- 3.3.3 Pressure sensitive adhesives approved as meeting these requirements shall be approved for use on all facestocks at an adhesive coating weight equal to or less than the coating weight specified in this protocol.
- 3.4 The process maps for testing a complete paper label laminate as supplied by a label manufacturer and for testing a PSA as supplied by an adhesive manufacturer are summarized below in Table 1.

Table 1. Process Map

Label Manufacturer, Converter, etc.	Adhesive Manufacturer	USPS-P-1238F Qualified Adhesive
Make label product	Make adhesive	
PSA laminate prep. Method A	PSA laminate prep. Method B	
Test via RCA LRP or optionally RCA MRP	Test via RCA LRP or optionally RCA MRP	
Certify through specification, verified by certified laboratory.	Certify through specification, verified by certified laboratory.	
Submit to TLMI for product listing	Submit to TLMI for adhesive listing	Submit to TLMI for adhesive and/or product listing

4. Requirements

- 4.1 Description – The paper label products and PSA’s covered by this protocol consist of components defined as the "face stock", "release liner backing" and "adhesive layer" or "PSA". The total construction may be referred to hereafter as the "laminate".
- 4.1.1 For the purposes of this testing protocol, the release liner is not specified since it typically is not a component in post consumer PSA labeling applications.
- 4.2 Face Stock – All label face papers must be made from chemical pulp (both virgin and recycled content).
- 4.2.1 Face stock paper cannot contain any ground wood or mechanical pulp.
- 4.2.2 The face stock papers identified below in Table 2 shall be used for “PSA Laminate Sample Preparation Method B”, for the qualification of a PSA as recycling compatible.

Table 2: Face Stock Requirements

Face Stock	Basis Weight (gram/m ²)
45 – 50 lb. Uncoated or electronic data processing (EDP) paper	66 - 81
45 – 50 lb. Thermal transfer paper	66 - 81
60 lb. C1S litho paper	84 - 93

4.3 Adhesive

- 4.3.1 The non-print side of the face stock shall be laminated with a PSA that shall enable the paper label to adhere and perform as required for its intended end use application.
- 4.3.2 The adhesion requirements of the PSA are independent of this protocol and are determined by the appropriate specification, if any, for adhesion performance in the specific paper label application.
- 4.4 PSA Laminate Sample Preparation Method A – This preparation method is intended for the qualification of finished paper label products as recycling compatible.
- 4.4.1 Manufacturers or converters of finished label products, seeking to obtain compliance under EO 13148 Sec. 702, shall produce the actual PSA laminate that is intended for use in a specific labeling application.
- 4.4.2 This laminate construction will be tested via the current version of RCA LRP or RCA MRP for conformance to the recycling compatible requirements as set forth in the RCA Specification.
- 4.4.3 Recyclability testing shall be done with a feed stock composition of 4.5% PSA label, 47.75% copy paper and 47.75% wove envelope paper.
- 4.4.4 Labels that do not have a continuous PSA coating over the whole paper surface will also be tested at the 4.5% PSA label level. Such samples must have the PSA coating weight equal to the average PSA coating weight of the label as it would be used in its intended application.

- 4.4.5 Laminates that are approved as meeting these requirements shall be approved only for the actual laminate construction and PSA at coating weights equal to or less than the coating weight submitted for testing. All other requirements for the PSA laminate shall be as specified in whatever performance specification applies to the end use labeling application for which the PSA laminate is intended.
- 4.5 PSA Laminate Sample Preparation Method B – This preparation method is intended for the qualification of a pressure sensitive adhesive as recycling compatible.
 - 4.5.1 Manufacturers seeking to obtain compliance under EO 13148 Sec. 702 for an adhesive shall produce PSA label laminates that are constructed from the adhesive being submitted for compliance on the three (3) face stocks specified in Table 2 of Section 4 above. If laboratory prepared samples of adhesive are being used, they must be in conformance with Section 3.3.1 of this protocol.
 - 4.5.2 Recyclability testing shall be done at a feed stock composition of 4.5% PSA label, 47.75% copy paper and 47.75% wove envelope paper. The 4.5% PSA label portion of this feedstock shall be a composite blend containing the following amounts (by weight) of these three (3) laminate constructions: uncoated or EDP paper @ 80parts; thermal transfer paper @ 10 parts and C1S litho paper @ 10 parts. The PSA coat weight on these laminates shall be 22 – 24 grams / sq. meter. This laminate composite blend will be tested via the current version of RCA LRP or alternatively via RCA MRP, for conformance to the recycling compatible requirements as set forth in the RCA Specification.
 - 4.5.3 Composite blends of these PSA laminates approved as meeting these requirements shall be approved for all paper label laminate constructions with a PSA coat weight of 24 grams / sq. meter or less.
 - 4.5.4 All other requirements for an adhesive shall be as specified in whatever performance specification applies to the end use labeling application for which a PSA laminate is intended.
- 4.6 Converter or PSA Manufacturer Submission Requirements – The following materials and information shall be supplied to the approved testing laboratory.
 - 4.6.1 Laminate samples with product codes.
 - 4.6.2 Label face stock paper with the supplier and product code.
 - 4.6.3 PSA supplier and product code.
 - 4.6.4 PSA coating weight in gm./m².
 - 4.6.5 Coating weight distribution across the web.
 - 4.6.6 Material Safety Data Sheets.

5. Equipment & Materials

- 5.1 Equipment
 - 5.1.1 Balance capable of weighing to 0.1 gram
 - 5.1.2 Thermometer
 - 5.1.3 pH meter
 - 5.1.4 Paper shredder capable of a ¼” strip cut (Model Privacy Guard, Medium Duty, 12-sheet strip cut or equivalent)
 - 5.1.5 Adirondack Formax Model 450H pulper
 - 5.1.6 Huygen slotted screen separator/analyzer (Somerville Screen) Model 1600 with 0.15 mm (0.006”) slots (or equivalent). An alternative is a Valley flat screen.
 - 5.1.7 Denver D12 laboratory flotation cell.
 - 5.1.8 105°C drying oven
 - 5.1.9 5-gallon plastic buckets
 - 5.1.10 Magnetic stirrer with stir bar
 - 5.1.11 Stopwatch or timer
 - 5.1.12 Assorted laboratory glassware
 - 5.1.13 Stand mixer (Lightning Model NAR-25 or equivalent)
- 5.2 Materials
 - 5.2.1 White copy paper, 20# weight, having a contaminant level of not greater than 5 ppm as measured by running the complete RCA LRP and RCA IAP on the copy paper alone.
 - 5.2.2 White wove envelope paper, 24# weight, having a contaminant level of not greater than 5 ppm as measured by running the complete RCA LRP and RCA IAP on the wove envelope paper alone. The wove envelope paper is available from the USDA Forest Products Laboratory, One Gifford Pinchot Drive, Madison, WI 53726. Web site is www.fpl.fs.fed.us/.
 - 5.2.3 1 N NaOH
 - 5.2.4 Soft water supply, control heated to 46°C

5.2.5 Aluminum foil or containers for drying screening rejects

6. Paper Feed Stock Preparation

6.1 Paper Feed Stock Preparation

- 6.1.1 Determine the moisture content for the copy paper, envelope paper and PSA laminate by weighing material before drying and after drying to a constant weight in a 105°C oven. Determine facepaper percent of PSA laminate by weighting a laminate sample and the facepaper with release liner removed from that same sample.
- 6.1.2 After determining the moisture content of materials and facepaper percent of PSA laminate, weigh to the nearest 0.1 g the appropriate amount of copy paper, envelope paper and PSA sample corrected for moisture content and for the PSA laminate, facepaper percent. The total oven dried (OD) weight of materials to be used is 360 g, comprised of the following: 171.9 g OD copy paper (47.75%); 171.9 g OD wove envelope paper (47.75%); and 16.2 g OD facepaper with adhesive (4.5%). The facepaper is typically attached to a release liner, therefore the facepaper percent of the laminate is needed to determine the correct amount of laminate to weigh out.
- 6.1.3 After all materials are weighed, adhere the facepaper to the envelope paper and allow adhesive bond development for a minimum of 72 hours before pulping.

7. Pulping

- 7.1 Shred all the paper into ¼" wide strips in the paper shredder. Shred the envelope paper with the adhered candidate label first.
- 7.2 Add 2040 g of soft water at $46 \pm 2^\circ\text{C}$ (115 F), adjusted to pH 10 with 1 N NaOH, to the pulper. (Caution: Wear eye and hand protection – NaOH is corrosive.) Use the auxiliary heater to maintain the pulper temperature at desired value and stir the water at 10 Hz.
- 7.3 Stop the stirring and measure the pH and temperature of the water before adding stock materials. Record on Data Sheet.
- 7.4 Add the shredded paper feed stock to the pulper. Allow the paper to thoroughly wet and ensure stock materials are not a solid clump.
- 7.5 Close the pulper lid and begin pulping at 10 Hz. Immediately increase to 18 Hz to prevent water loss through the lid seal.
- 7.6 Increase the frequency to 40 Hz as soon as possible. Stop the pulper just after reaching 40 Hz and push any material down off of the pulper wall without adding water. Restart the pulper at 40 Hz, start the stopwatch and continue pulping for a total of 8 minutes.
- 7.7 After the 8-minute pulping time, turn off the pulper. Measure the pH and temperature of the pulp. Record on the Data Sheet.
- 7.8 Deactivate the pulper power supply.
- 7.9 Drain the pulper contents into a 5-gallon plastic bucket.
- 7.10 Mix the pulp in the bucket thoroughly by hand and then collect a sample for consistency (% solids) measurement. Measure the consistency of the pulp by drying a sample in the 105°C oven until a constant weight is achieved. Record on the Data Sheet. Target consistency is 15% csc.
- 7.11 Take 30 g OD pulp (200 g at 15% csc) and place in a separate 5-gallon bucket. Dilute this material to 1% csc by adding 2,800 g water to the 200 g sample at 15% csc. The 1% pulp will be used for making handsheets. Mix the 1% sample with a stand mixer for at least 5 minutes to ensure homogeneity prior to handsheet making.
- 7.12 Make 16 handsheets according to RCA IAP.
- 7.13 Clean the pulper.
- 7.14 Save the remaining pulp for screening analysis.

8. Slotted Screening

- 8.1 Prior to starting, ensure that a 6-cut screen is installed in the Huygen Somerville Screen.
- 8.2 The accepts are collected in a 150 mesh screen box (the typical screening yield is approximately 75%). The rejects are any deposits left in the doser tank or screening chamber.
- 8.3 Adjust the mode to "Demo" on the keypad and run at least 2 cleaning cycles to flush the system of residual cool water and manually adjust the screening water temperature to $46 \pm 2^\circ\text{C}$.
- 8.4 Ensure that the Calibrated Flow Nozzle rate is 8.6 Lpm and the Doser Spray is an additional 3.9 Lpm for a total of 12.5 Lpm total on the flow meter.

- 8.5 Adjust the mode to “Stickies” on the keypad and begin the automated screening process.
- 8.6 From the remaining pulper material, take 200 g OD of pulp (1333 g at 15% csc). Place the pulp in the doser tank of the Somerville Screen as an even layer of pulp (gently level out the pulp by hand).
- 8.7 Allow the program to run to completion (approximately 38 minutes).
- 8.8 After the automated screening process is complete, collect the accepts in a 5-gallon bucket and collect the rejects in a pre-weighed aluminum container for oven drying at 105°C.
- 8.9 Clean all sections of the screen unit.
- 8.10 After screening, take 30 g OD of the screening accepts and place in a separate 5-gallon bucket to be used for handsheet analysis. For exactly 10% csc, 30 g OD would be 300 g of wet pulp. Dilute this material to 1% csc (add 2700 g water to 300 g sample at 10% csc) in preparation for making handsheets. Mix the 1% sample with a stand mixer for at least 5 minutes to ensure homogeneity prior to handsheet making.
- 8.11 Make 16 handsheets according to IAP.
- 8.12 With the remaining screening accepts, perform flotation analysis.

9. Flotation

- 9.1 Prepare a 0.5% DI-700 surfactant solution by weighing 5 g of 100% surfactant in a 1000 mL glass beaker. Add approx. 995 mL of hot water into the beaker and stir with a magnetic stirrer until the DI-700 dissolves in water.
- 9.2 In the Denver D12 laboratory flotation cell, place 90 g OD of pulp (approx. 900 g at 10% csc) and add $46 \pm 2^\circ\text{C}$ water to achieve a total volume of 9 liters for a 1% pulp csc.
- 9.3 Add 18 mL of 0.5% DI-700 surfactant solution, equivalent to approx. 0.1% loading based on fiber dry weight, and then turn on the rotor to mix the pulp and surfactant for 1 minute.
- 9.4 Run the Denver D12 laboratory flotation cell for five minutes with the airflow set at the fully open position. Wait one minute, and then periodically scrape the top layer of foam from the cell during the remaining four minutes.
- 9.5 After the five-minute run time, stop the airflow, measure and record the temperature. Save the accepts in a 5-gallon bucket for handsheet analysis.
- 9.6 Collect the rejects from the sidewall of the flotation cell and overflow receptacle. Dewater the rejects on a weighed filter paper with a Büchner funnel, making a pad. Oven dry the pad and determine the net weight in grams and percent rejects, and reject rate in grams per minute.
- 9.7 Mix the 1% sample with a stand mixer for at least 5 minutes to ensure homogeneity prior to handsheet making.
- 9.8 Make 16 handsheets from the accepts pulp according to the image analysis procedure (RCA IAP).
- 9.9 Clean the flotation unit.

10. Handsheet Preparation and Analysis

- 10.1 For each of the pulp samples (pulper, screening, and flotation) mix the diluted pulp with a stand mixer for at least 5 minutes to ensure homogeneity immediately prior to handsheet making.
- 10.2 For each unit operation make 16 handsheets according to the procedure in RCA IAP.

11. Reporting

- 11.1 The report shall consist of the following:
 - 11.1.1 Copies of Data Log Sheets
 - 11.1.2 Weight of rejects from the 6 cut screen and flotation.
 - 11.1.3 Image analysis data including equipment settings, number of hand sheets analyzed, mean values for PPM, counts per square meter, PPM 95% confidence interval, average particle size, and the removal efficiency of each operation.
 - 11.1.4 A mass balance analysis for the adhesive, if possible.

Addendum 1
RCA-LRP

Verification Form – Recycling Compatible Label or PSA Product

Certified Testing Laboratory Information:

Name: _____
Address: _____

Primary Contact(s): _____
Phone No. _____ Fax No. _____
E-Mail: _____

Type of verification (check one)

- ☐ Qualification of a specific PSA laminate or label as recycling compatible
☐ Qualification of a specific PSA as recycling compatible

PSA manufacturer or converter supplied information

Name: _____
Address: _____

Primary Contact(s): _____
Phone No. _____ Fax No. _____
E-Mail: _____

Feedstock for recyclability testing

Sample Information	1 Finished Label	2	3	4
Name of PS adhesive tested				
Code no. of PS adhesive tested				
Name of PS laminate tested				
Code no. of PS Laminate tested				
Face stock type per sample preparation Method A or B	Identify here	Uncoated EDP paper	Thermal transfer paper	C1S litho paper
Face stock basis wt. Gm./m ²				
Sample loading, Gm.				
Sample loading, %				
Envelope paper loading, Gm.				
Envelope paper loading, %				
Copy paper loading, Gm.				
Copy paper loading, %				

Coating weight distribution across the web (uniform coating, stripe coating, etc.) _____

Run report

Copies of Data Log Sheets

Image analysis data including equipment settings, number of hand sheets analyzed, mean values for PPM, counts per meter², PPM standard deviation, average particle size, and efficiency of each operation.

A mass balance analysis for the adhesive, if possible.

Addendum 1 (Continued)
RCA-LRP

Verification:

I (we) verify that the PSA laminates submitted for recyclability testing were tested according to the RCA Laboratory Recycling Protocol, RCA LRP, and that the laminates passed the following specifications as defined in the Specification for Paper Labels coated with recycling compatible Pressure Sensitive Adhesives.

1. Average adhesive particle size after pulping was greater than 0.23 mm².
2. Average adhesive area after screening was less than 430 ppm.
3. Average adhesive area after flotation was less than 10 ppm.
4. Average residual contamination in the blank copy paper was less than 5 ppm.
5. Average residual contamination in the blank white wove envelope paper was less than 5 ppm.

Signature_____

Title_____

Certified Laboratory_____

Date_____