(The following is a test protocol for a "tea bag analysis" that was conducted on PMN starches by the Corn Refiners Association. EPA has been granted permission to disseminate this protocol by the National Starch and Chemical Co. This protocol is one approach to providing information on swellability of high molecular weight polymers and EPA realizes that often there are a number of different but acceptable means to providing testing information. Before starting to conduct any study, the PMN submitter must obtain approval of test protocols from EPA by submitting written protocols. EPA's acceptance of a particular protocol depends on multiple factors including the specifics of the test substance, purpose of the testing, familiarity with specific procedures and equipment, validation of the method, etc.)

MOISTURE ABSORPTION/RETENTION

DRY STARCH POWDERS
Modified Teabag Method

Introduction: This test is a measure of the quantity of water (saline solution) which can be absorbed and retained by a granular starch. The starch is sealed in a teabag, immersed in water, drained, and the weight recorded (absorbent capacity). The teabag is then centrifuged and reweighed (retentive capacity).This procedure has been modified to account for starch lost through the teabag fabric.

Equipment:
Temperature Bath (37°C)
Heat Sealer (Semcorp Systems)
Centrifuge (Marathon 21K/R, Fisher Scientific) and tubes (60 ml)
Analytical Balance (20g min, 0.0001g)
Timer
Beakers - 2000 ml (2)
Paper Clips
pH Meter

Materials:
Dexter Nonwoven Grade 7291 Fabric
Kimwipe EX-L Tissues
Paper Towels
0.9% Saline Solution (Reagent Grade Sodium Chloride in Deionized Water)
Hydrochloric Acid Solution (1N)
Sodium Hydroxide Solution (1N)

Procedure:

1. Fill two 2000 ml beakers with 1000 ml of 0.9% saline solution. Adjust the pH of the saline solution to 5.1-5.3 and 7.1-7.3 respectively by adding HCl and/or NaOH solution to the beakers.
2. Place the beakers in the temperature controlled water bath (37°C) and allow to

stand until equilibrated (30 minutes minimum).

3. Construct 3 teabags from Dexter nonwoven fabric (grade 7291).
   a. Using a paper cutter cut fabric to a standard size (15.4cm x 7 cm)
   b. Fold in half in the short direction
   c. Heat seal a 10 mm wide strip along both edges (140°C/40 psi/4 sec). With a pencil write an ID number on the bags.
   d. Make a small hole with a paperclip through the top (unsealed edge of each bag.
4. Weigh (0.0001g) each empty bag (W_{empty}).
5. Accurately weigh (0.0001g) into each teabag approximately 0.20 g of starch (W_{starch}). The moisture content (%M) of each starch must be independently determined (CML 116B or equivalent).
6. Seal the top of each bag just below the hole and insert a paperclip hanger into each bag.
7. Hang all bags, completely immersed, in the appropriate thermostated saline solution.
8. After the indicated immersion time (10 or 60 minutes) carefully remove the bags from the solution , remove the paperclip and drain each on a separate paper towel for 10 minutes.
9. Weigh (0.0001g) and record each wet teabag ($W_{immersion}$).
10. Place two folded Extra Large Kimwipes and one tea bag in a centrifuge tube and spin for 10 minutes at ≈3000 RPM.
11. Weigh (0.0001g) and record each spin-dried teabag (W_{cent}).
12. Carefully place each bag in a forced air oven (100-110°C) for 6-10 hrs.
13. Remove the dry teabags from the oven, cool in a desiccator and quickly weigh them (0.0001g, W_{dry}).

Blanks:

For each set of samples analyzed, 3-6 blank teabags should be run. Repeat steps 3 - 13 but do not add starch to the bags. For each of the blank bags calculate $\mathbf{W_1}$, $\mathbf{W_2}$ and $\mathbf{W_3}$ and the average of each of these constants ($\mathbf{W_{1avg}}$, $\mathbf{W_{2avg}}$ and $\mathbf{W_{3avg}}$).

$$\mathbf{W_1 = W_{empty} - W_{dry}}, \quad \mathbf{W_2 = W_{immersion} - W_{dry}}, \quad \mathbf{W_3 = W_{cent} - W_{dry}}.$$

Calculations:

$$Absorbent\ Capacity = \frac{W_{immersion} - W_{dry} - W_2}{W_{dry} - (W_{empty} - W_1)}$$

$$Retentive\ Capacity = \frac{W_{cent} - W_{dry} - W_3}{W_{dry} - (W_{empty} - W_1)}$$

Reporting:

Report the average and the sample standard deviation of both the absorbent and retentive capacities. Ignore values for teabags with greater than 10% starch loss.

Notes and Comments:

$$Starch\ Loss\ (\%) = 100 \times \left(1 - \frac{\%M \times (W_{dry} - (W_{empty} - W_1))}{W_{starch} \times (100 - \%M)}\right)$$

1. Some starch migrates through the teabag fabric. To minimize this loss the teabags (both wet and dry) must be handled gently and with care. A number of fabric grades (Dexter 7291, 11521 and 1189) were examined for this analysis. None were found to be significantly better than the specified Dexter 7291.

2. A Lotus 123 spreadsheet template has been setup for these calculations.