Grease resistance test for paper and paperboard

**CAUTION:** This method may require the use, disposal, or both, of chemicals which may present serious health hazards to humans. Procedures for the handling of such substances are set forth on Material Safety Data Sheets which must be developed by all manufacturers and importers of potentially hazardous chemicals and maintained by all distributors of potentially hazardous chemicals. Prior to the use of this test method, the user should determine whether any of the chemicals to be used are potentially hazardous and, if so, must follow strictly the procedures specified by both the manufacturer, as well as local, state, and federal authorities for safe use and disposal of these chemicals.

1. **Scope and significance**

   1.1 This new provisional method is an expanded and updated version of TAPPI UM 557 “Repellency of Paper and Board to Grease, Oil, and Waxes (Kit Test).”
   1.2 Commonly known as the kit test, the method describes a procedure for testing the degree of repellency and/or the antiwicking characteristics of paper or paperboard treated with fluorochemical sizing agents (1,2).
   1.3 Fluorochemical agents may impart both organophobic and hydrophobic characteristics to paper through a reduction in the surface energy of the sheet. This is done by a surface treatment of the fibers without the formation of continuous films.
   1.4 This test was originally developed to allow papermakers to know when the applied fluorochemical was incorporated into the sheet and the approximate level of resistance. These were accomplished by testing production samples with a series of numbered reagents (varying in surface tension and viscosity or “aggressiveness”) held in bottles in a specially designed kit. The highest numbered solution (the most aggressive) that remained on the surface of the paper without causing failure was reported as the “kit rating” (hence the term “kit test”). This concept forms the basis for the current provisional method.
   1.5 Designed primarily as a surface repellency test for fluorochemical treatments, the use of this test for papers with film-like barriers or those treated with nonfluorochemical agents would have to be evaluated.
   1.6 A related test is TAPPI T 454 “Turpentine Test for Voids in Glassine and Greaseproof Papers” which is more limited in terms of paper type and may detect a different failure mechanism than the current method.

2. **Apparatus**

   2.1 *Absorbent tissue or cotton*; to wipe away kit solutions at end of test.
   2.2 *Balance*, capable of weighing 4000 g to within 0.1 g.
   2.3 *Graduated cylinders*, glass, 100 and 500 mL; for preparing kit solutions.
   2.4 *Stock bottles*, glass with foil-lined caps, glass stoppers, or polyethylene-lined caps to prevent evaporation losses; for storing kit solutions.
   2.5 *Stopwatch or timer*.
   2.6 *Test bottles*, with glass stoppers and glass droppers; for use during testing.
2.7 **Testing area**, e.g., well-lit bench with dark top located in standard-conditions testing room; for conducting test(s) and rating results.

3. **Reagents**

3.1 *Castor oil.*
3.2 *n-heptane.*
3.3 *Toluene.*
3.4 *Kit solutions.*
3.4.1 Prepare the kit (test) solutions from the above reagents using the mixtures shown in Table 1.

**CAUTION:** Consult appropriate Material Safety Data Sheets and applicable safety requirements and regulations before working with n-heptane and toluene. Both are highly flammable and are reported to be health hazards. Keep away from heat and sparks and avoid prolonged or repeated breathing of vapors and contact with skin.

3.4.2 Do not measure the reagents by addition since there will be a loss of volume after mixing.
3.4.3 Weight measurements are used for castor oil as its high viscosity makes volume measurements less accurate. The density of castor oil is 0.969 g/cm³.
3.4.4 Store the solutions in labeled stock bottles. As required, fill each test bottle with the appropriate kit number solution from the stock bottles.
3.4.5 The stock and test bottles should be tightly closed when not in use to prevent loss of solvent from evaporation (and a consequent change in the composition and properties of the solution).
3.4.6 The solutions in the test bottles should be changed regularly depending on usage. For frequent daily usage, solutions should be replaced weekly with fresh solution from the stock bottles; for less frequent usage, a monthly solution change is adequate.

**NOTE 1:** For certain applications, it has been reported that the upper limit of the range of kit ratings has been extended beyond 12 by continuing to adjust the proportion of toluene and n-heptane. The validity of doing this is uncertain. If done, the report should state clearly the ratio of toluene to n-heptane used to arrive at the extended kit values.

4. **Safety precautions**

4.1 As previously noted, various hazards including (but not limited to) flammability, explosivity, and toxicity, singly or in combination, are reportedly associated with reagents which may be used in this method.
4.2 It is the responsibility of the user to determine current applicable requirements and regulations which may be in force for the materials used at the time this method is applied. Those requirements and regulations take precedence where they are, or appear to be, in conflict with any of the provisions in this document.
4.3 General precautions include (but are not limited to): avoidance of an open flame in any part of this test and the use of approved safety goggles, gloves, lab coat, and ventilated hood.

5. **Sampling**

5.1 The method used to obtain samples for testing depends on the scope and intent of the sampling program. Sample the paper or paperboard to be tested in a manner which has been agreed to in advance.
Table 1. Mixtures of reagents for preparing kit solutions.

<table>
<thead>
<tr>
<th>Kit No.</th>
<th>Castor Oil, g</th>
<th>Toluene, mL</th>
<th>n-heptane, mL</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>969.0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>2</td>
<td>872.1</td>
<td>50</td>
<td>50</td>
</tr>
<tr>
<td>3</td>
<td>775.2</td>
<td>100</td>
<td>100</td>
</tr>
<tr>
<td>4</td>
<td>678.3</td>
<td>150</td>
<td>150</td>
</tr>
<tr>
<td>5</td>
<td>581.4</td>
<td>200</td>
<td>200</td>
</tr>
<tr>
<td>6</td>
<td>484.5</td>
<td>250</td>
<td>250</td>
</tr>
<tr>
<td>7</td>
<td>387.6</td>
<td>300</td>
<td>300</td>
</tr>
<tr>
<td>8</td>
<td>290.7</td>
<td>350</td>
<td>350</td>
</tr>
<tr>
<td>9</td>
<td>193.8</td>
<td>400</td>
<td>400</td>
</tr>
<tr>
<td>10</td>
<td>96.9</td>
<td>450</td>
<td>450</td>
</tr>
<tr>
<td>11</td>
<td>0</td>
<td>500</td>
<td>500</td>
</tr>
<tr>
<td>12</td>
<td>0</td>
<td>450</td>
<td>550</td>
</tr>
</tbody>
</table>

5.2 If the testing is being done to determine the properties of a lot (for instance, to determine whether the lot meets specifications), take a sample in accordance with T 400 “Sampling and Accepting a Single Lot of Paper, Paperboard, Containerboard, or Related Product.” For other specialized purposes, take a representative sample.

6. Test specimens

6.1 From each test unit of the sample, cut five test specimens at least 51 mm x 152 mm (2 in. x 6 in.). Standard sheets cut 216 mm x 279 mm (8½ in. x 11 in.) are an easy size to work with; if both sides are to be tested, these sheets can be cut in half in the long direction to make a set of paired (felt side/wire side) specimens. Label or identify in some manner each side of the test specimens. Since paper and paperboard may be treated on one side or both sides, or the treatment may be different between sides, depending on the end use, the tester must decide which side(s) to test. A fluorochemical treatment applied uniformly to both sides of a paper may differ by as much as one kit value, depending on the difference in fines content between the two sides.

6.2 Use care in handling the test specimens. The use of plastic disposable gloves to protect the sample is recommended. Do not test in an area that has fingerprints, oil from a previous test, an abraded surface, wrinkles, or scores.

7. Conditioning

Precondition (on the dry side), condition, and test specimens in atmospheres in accordance with TAPPI T 402 “Standard Conditioning and Testing Atmospheres for Paper, Board, Pulp Handsheets, and Related Products.”

8. Procedure

8.1 Select an area of the testing lab that is clean and well illuminated. A laboratory bench with a dark gray top works well and provides contrast for viewing the tested paper. The lighting should be uniform and consistent in quality over time.

8.2 Place each test specimen on a clean, flat surface, test side up.

8.3 Select a specimen to be tested and back it with a darker material if a dark benchtop is not available.

8.4 Select an intermediate kit number test solution (test bottles). From a height of about 13 mm (0.5 in.), gently release a drop onto the surface of the test specimen, and start the timer. Be careful not to touch the glass eyedropper to the surface of the paper.

8.5 After 15 s, quickly remove the excess test solution with a clean tissue or cotton swatch, and immediately examine the test area. A failure is denoted by a darkening of the test specimen, even in a small area. (The darkening results from wetting of the specimen due to penetration of the test solution.) If the tested area is not examined immediately, volatile components of the test solution will be lost and the area may regain its original light reflectance value and be scored as “passed”.

8.6 If the specimen fails this first test, select an untested area on the same test specimen and repeat the test using the next lower numbered kit solution. Repeat until the highest numbered kit solution that rests on the surface for 15 s without causing failure is identified. If the specimen passes this first test, repeat the test on an untested area using a higher numbered kit solution. Repeat until the highest numbered kit solution that does not cause failure is identified. The number of this kit solution is the kit rating for the specimen.
8.7 Repeat the procedure in 8.6 for the remaining four test specimens and record each of their kit ratings. (Results from the first test can be used as an indication of which kit solution to use for the first drop on these remaining four specimens.)

9. Calculation

Calculate the arithmetic average of the five determinations (kit ratings) for each unit of paper or paperboard tested.

10. Report

10.1 Report the test value as the arithmetic-average kit rating to the nearest 0.5 units.

10.2 Report the number of determinations per test value and the standard deviation (or minimum and maximum).

10.3 State clearly and conspicuously any deviations from the standard procedure (see Note 1), and note any unusual features of the sample.

11. Precision

11.1 The average maximum expected difference between two test results, each of which is the average of five test determinations, is:

Repeatability (within a laboratory) = 0.7 kit rating units or 9%.

Table 2. Precision (repeatability) data for “Grease Resistance Test for Paper and Paperboard” (Kit Ratings).

<table>
<thead>
<tr>
<th>Determination No.</th>
<th>Paper 1</th>
<th>Paper 2</th>
<th>Paper 3</th>
<th>Paper 4</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>11</td>
<td>6</td>
<td>7</td>
<td>8</td>
</tr>
<tr>
<td>2</td>
<td>9</td>
<td>8</td>
<td>7</td>
<td>8</td>
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<tr>
<td>3</td>
<td>9</td>
<td>7</td>
<td>7</td>
<td>8</td>
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<tr>
<td>4</td>
<td>9</td>
<td>6</td>
<td>7</td>
<td>8</td>
</tr>
<tr>
<td>5</td>
<td>11</td>
<td>7</td>
<td>8</td>
<td>8</td>
</tr>
<tr>
<td>Ave. 9.8</td>
<td>6.8</td>
<td>7.2</td>
<td>8</td>
<td></td>
</tr>
<tr>
<td>Std. Dev.</td>
<td>1.0954</td>
<td>0.8367</td>
<td>0.4472</td>
<td>0</td>
</tr>
<tr>
<td>Std. Dev. of Test Result</td>
<td>0.4899</td>
<td>0.3742</td>
<td>0.2000</td>
<td>0</td>
</tr>
<tr>
<td>Repeatability, r</td>
<td>1.36</td>
<td>1.04</td>
<td>0.55</td>
<td>0</td>
</tr>
<tr>
<td>Repeatability, %</td>
<td>13.8</td>
<td>15.2</td>
<td>7.7</td>
<td>0</td>
</tr>
<tr>
<td>Ave. r</td>
<td></td>
<td>0.74</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ave. r%</td>
<td></td>
<td>9.19</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Reproducibility (between laboratories) = not known.

11.2 Repeatability value was determined in accordance with the definitions of this term in TAPPI T 1206 “Precision Statement for Test Methods” and is the average value for four commercially made papers or paperboard obtained in one laboratory by an experienced tester (see Table 2).

NOTE 2: The standard deviation of a test result was calculated by dividing the standard deviation by the square root of 5.

12. Keywords

Fluorine compounds, size, grease resistance, oil, repellence, paper, paperboard.

13. Additional information

13.1 Effective date of issue: February 16, 1996.

13.2 This new Provisional Method is an expanded update of TAPPI UM 557 “Repellency of Paper and Board to Grease, Oil, and Waxes (Kit Test).” Due to the extensive use of this test in the paper industry, and because TAPPI
Useful Methods will no longer be published, request was made to the Physical Properties Committee to develop a Provisional Method based on TAPPI UM 557. The current draft lacks reproducibility data.

13.3 Detection of failure is sometimes difficult in “tight” sheets (those that are highly refined and have high apparent density and/or low air permeability) and “thin” sheets (those with low thickness and low opacity). Backing these sheets with a darker material, such as a dark bench top, may be helpful in deciding if the test specimen has passed or failed a kit test.

13.4 Fines and fillers may preferentially attract some fluorochemicals. Thus, the two sides of a sheet having marked two-sidedness may differ by as much as one kit unit. This should be considered in devising a sampling and testing plan.

13.5 Related methods: TAPPI UM 557.

Literature cited


Your comments and suggestions on this procedure are earnestly requested and should be sent to the TAPPI Technical Divisions Administrator.