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T \_\_\_\_\_ 454 \_\_\_\_\_

DRAFT NO. \_\_\_\_\_ 02-SARG \_\_\_\_\_

DATE \_\_\_\_\_ May 18, 2021 \_\_\_\_\_

WORKING GROUP  
CHAIRMAN \_\_\_\_\_ To be determined \_\_\_\_\_

SUBJECT  
CATEGORY \_\_\_\_\_ Physical Properties \_\_\_\_\_

RELATED  
METHODS \_\_\_\_\_ See "Additional Information" \_\_\_\_\_

**CAUTION:**

This Test Method may include safety precautions which are believed to be appropriate at the time of publication of the method. The intent of these is to alert the user of the method to safety issues related to such use. The user is responsible for determining that the safety precautions are complete and are appropriate to their use of the method, and for ensuring that suitable safety practices have not changed since publication of the method. 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 method, the user must determine whether any of the chemicals to be used or disposed of 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.

**Turpentine test for voids in  
glassine and greaseproof papers  
(Five-year review of Official Method T 454 om-15)  
(No changes from previous draft: Standard reaffirmed)**

**1. Scope and significance**

This method gives an accelerated comparison of the relative rates at which oils or greases, such as commonly found in foodstuffs, may be expected to penetrate papers such as greaseproof, glassine, and vegetable parchment. In addition, it may be used to select and predict the performance of these grades of papers for an intended end use. The selection should be used as preliminary to, and not a substitute for, tests with prototype end products containing the oils or greases of interest. It may not be applicable to grades of paper or paperboard that are given grease or oil resistance by means of a coating or internal treatment.

## 2. Apparatus

2.1 *Tube*, of any rigid material, 25 mm (1 in.) internal diameter and at least 25 mm (1 in.) in height, the ends of which have been smoothed.

2.2 *Buret* or automatic pipet, calibrated to deliver 1.1 mL of liquid.

2.3 *Sand*, Ottawa grade sand, a spherical, natural silica, sand used for cement testing, available from laboratory supply houses, screened to pass a No. 20 mesh screen and be retained on a No. 30 mesh sieve.

2.4 *Paper*, white coated and calendered sheets of book paper, 104 g/m<sup>2</sup> (70 lb 25 × 38 - 500) of convenient size.

2.5 *Timing device*, stopwatch or laboratory timer.

2.6 *Watch glass*, 7.6 cm diameter.

2.7 *Scoop*, 5-g capacity; verify the capacity of the scoop by weighing a few scoop charges of sand on an analytical balance; each charge should weigh  $5.0 \pm 0.1$  g and be consistent.

## 3. Reagent

*Turpentine*, moisture-free and colored; to 100 mL of pure gum spirits turpentine, sp gr 0.85 to 0.91 at 16°C (60°F), add 5 g of anhydrous calcium chloride and 1.0 g of an oil-soluble red dye. Stopper the container, shake well, and let stand for at least 10 h, shaking occasionally. Then filter through a dry filter paper at room temperature, and store in an airtight bottle. Use pure artist, or a clear grade of turpentine. Lower purity grades of turpentine that can be bought in paint stores may be cloudy or yellow in color and are not suitable for this test.

## 4. Sampling and test specimens

From each test unit obtained in accordance with TAPPI T 400 "Sampling and Accepting a Single Lot of Paper, Paperboard, Containerboard, or Related Product," prepare ten 100 mm (4 in.) square specimens identifying five as felt (top) side and five as wire (bottom) side.

## 5. Procedure

5.1 Condition and test the specimens in an atmosphere in accordance with TAPPI T 402 "Standard Conditioning and Testing Atmospheres for Paper, Board, Pulp Handsheets, and Related Products."

**NOTE 1:** It has been determined that temperature has a strong influence on the test results.

5.2 Place each test specimen on a portion of a sheet of coated book paper which has been positioned on a smooth, flat horizontal surface such that no portion of the specimen extends beyond the book paper. Place the tube (2.1) on end on the specimen and with the scoop put  $5 \pm 0.1$  g of the sand in the tube. Because the purpose of the tube is solely to ensure a uniform area of the sand pile, remove the tube immediately after the addition of the sand by carefully lifting it straight up.

5.3 Add 1.1 mL of the colored turpentine to the sand pile using the calibrated buret or automatic pipet. The 1.1 mL of turpentine will saturate exactly 5 g of sand. Start the timing device as the last drop of turpentine leaves the pipet or burette.

5.4 Test nine more specimens, four with the same surface up as with the first specimen, five with the opposite surface. When this is done sequentially, start the timing device immediately after the turpentine has been added to the first specimen. Now repeat 5.2 and 5.3 for each specimen at equally incremental times (e.g., every 10 s) to facilitate recording the end point. The test sequence begins at the moment of saturation of the sand and ends when staining of the book paper is observed.

5.5 At periodic intervals, gently slide each specimen to a new unexposed portion of the book paper and examine the previously exposed portion for the staining which indicates that the turpentine has penetrated the specimen. It is advisable to make a few preliminary tests to determine the approximate end point. From this the correct intervals for examination can be determined. As a minimum, it is suggested that for the initial tests to make observations every 15 seconds for the first minute, then at 1-minute intervals for the next 5 minutes, and finally every 5 minutes until the maximum time is reached at 30 minutes.

**NOTE 2:** Cover with a watch glass any specimen which requires over 2 min (120 s) to stain.

5.6 Record the elapsed time from addition of the turpentine to the sand to the first sign of staining of the book paper. If staining has not occurred in 30 min (1800 s), terminate the test.

## **6. Report**

6.1 Report the test result in seconds, as the average of ten determinations, five on the wire side and five on the felt side, to two significant figures. When one or more determinations exceeding 1800 s are included in the test result, report the average followed by a plus sign.

6.2 The following are two examples of the recommended form:

6.2.1 *High-range turpentine test specimen, s*

<i>Test</i>	<i>Wire side</i>	<i>Felt side</i>
1	540	780
2	660	1800+
3	1800+	1800+
4	1800+	1800+
5	1800+	1800+

Test result (= average over 10 specimens): 1500+

6.2.2 *Medium-range turpentine test, s*

<i>Test</i>	<i>Wire side</i>	<i>Felt side</i>
1	960	780
2	780	540
3	540	420
4	960	180
5	180	1320

Average (test result), 670

**7. Precision**

## 7.1 Repeatability (within a laboratory):

Low range (30-300 s): 60%

Medium range (300-900 s): 68%

High range (above 900 s): not known

## 7.2 Reproducibility (between laboratories):

Low range: 84%

Medium range: 82%

High range: not known

7.3 The terms “repeatability” and “reproducibility” are defined in TAPPI T 1200 “Interlaboratory Evaluation of Test Methods to Determine TAPPI Repeatability and Reproducibility.” The calculations are based on data obtained in a 1972 Glassine and Greaseproof Division API interlaboratory study in which six laboratories participated, testing one material at each of the three ranges. For each material each laboratory obtained five test results, each test result being the average of five determinations on each side of the materials. Most of the high range readings were at the test limit (1800+).

## **8. Keywords**

Paper, Glassine papers, Greaseproof papers, Oil penetration tests, Grease resistance, Voids, Parchment papers

## **9. Additional information**

9.1 Effective date of issue: To be assigned.

9.2 Reference: TAPPI Subcommittee on Grease Resistance (1).

9.3 Related method: PAPTAC F.6.; ISO 16532-3

9.4 This method was revised in 1944, 1960, 1966, 1973, 2000, and 2010. The 1977 version contained precision data as described in 7.3, but with some test results eliminated. In 1984 the precision statement was recalculated from all of the data, producing the 1989 revision. The 2000 revision consisted of a change in title and scope and clarification of the description of the derivation of the precision statement. The 2010 revision clarified the description of the sand and the turpentine and clarified the timing of the procedure in 5.5.

## **Reference**

1. TAPPI Subcommittee on Grease Resistance, “Suggested Method for Testing Paper for Grease Resistance,” *Paper Trade J.* **103**(26):20 (1936).

*Your comments and suggestions on this procedure are earnestly requested and should be sent to the TAPPI Standards Department.* ■