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T \_\_\_\_\_ 657 \_\_\_\_\_

BALLOT NO. \_\_\_\_\_ 02-SARG \_\_\_\_\_

DRAFT NO. \_\_\_\_\_ 01 \_\_\_\_\_

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WORKING GROUP  
CHAIR \_\_\_\_\_ N/A \_\_\_\_\_

SUBJECT  
CATEGORY \_\_\_\_\_ Coating and Graphic Arts \_\_\_\_\_

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 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.

## **Sampling of Fillers and Pigments**

*(Five-year review of Standard Practice T 657 sp-21)*

### **1. Scope**

This document describes procedures for sampling shipments of fillers, pigments, and other materials in finely divided form for the purpose of securing a sample for analysis. Procedures are given for sampling dry bulk and bagged shipments, as well as high-solids slurries.

### **2. Significance**

2.1 This standard describes procedures to sample shipments of particulate material, notably pigment and filler material and either dry or in suspension, in an indicative and consistent manner.

2.2 Such particulate material should be already in the form and fineness required for its application. Typically this means: In the form of dry powder or suspensions of micron sized, disperse particles.

2.3 Any larger particles or lumps should then be only the result of caking or compaction for powders - or respectively of settling or crust formation for suspensions.

2.4 The described equipment and procedure, described below, are typically not suitable for sampling of materials, which contain or even consist of lumps, agglomerates or pebbles equal to or larger than ca. 1/8 inch.

### 3. Apparatus

3.1 *For dry bulk shipments: Bulk sampler*, commonly known as a grain sampler or thief, consisting of a probe having two close-fitting concentric tubes with open slots along the sides and ending in a cone, with the tubes and openings large enough to accommodate the largest lumps in the material to be sampled. Tube diameter and opening of about 35 mm (1½ in.) should be satisfactory for material containing lumps no larger than **12mm (0.5 in.)** (P.Gy, 1982). Samplers of 1.67 m (66 in.), 1.83 m (72 in.), and 3.05 m (120 in.) length of the type recommended by the Association of Official Analytical Chemists are available if desired.

3.2 *For dry bag, drum, or packaged shipments: "Thief,"* a simple and inexpensive device fabricated from a length of thin-walled rigid plastic pipe about 150 mm (6 in.) longer than the width of the containers to be sampled and about 38 mm (1.5 in.) in outside diameter. For sampling of fine powder materials, i.e. <100micron, not containing larger particles, one can use a 'thief' of lower diameter, which can be easier inserted into dry bags and similarly packaged shipments, i.e. by pushing it through the bag lining and sealing the hole after sampling with tape. E.g. a 'thief' of ½ to ¾ inch diameter is commonly used to sample 1 ton big bags. The pipe is sawed in half lengthwise from one end to about 150 mm from the opposite end and cut perpendicular in this position to remove one of the long sections. The other end of the remaining long section is ground or filed to a gentle point to permit easy insertion through the container filling valve into the contents of the container for sampling.

3.3 *For high-solids slurries in tank truck: Wide mouth plastic bottle*, of approximately 1000 or 2000 mL capacity, having a tight-fitting rubber stopper through the center of which a length of light rope is firmly affixed, taped to one end of a handle (rod) of sufficient length to permit sampling at the maximum desired depth. The sampling bottle is positioned parallel to the handle with the mouth facing towards the far end (top) of the handle. When used, the stopper is put firmly into place, the sampling bottle submerged to the desired depth, and the stopper pulled free from above to permit the bottle to fill.

### 4. Procedure

**NOTE 1:** Sampler or sample containers should be clean and should be cleaned between each sample taken.

4.1 If possible, sample the shipment as it is being delivered. Sample at predetermined or random intervals during unloading, so that the number of increments sampled will be in accordance with that specified in 4.2, 4.3, or 4.4. Label or mark sample containers indicating minimally a) product sampled, b) date & time of sampling and c) name of the person conducting the sampling. For hazardous products, foresee the sample container with the appropriate product safety label.

4.2 *For bulk shipments, as in bins or piles*

4.2.1 Sample a sufficient number of portions of the shipment as to be representative, e.g., a number increasing with the size of the shipment, as in Table 1 with a carload being considered equivalent to 501 to 1300 "units." If a carload is shipped in bulk and cannot be sampled during unloading as specified in 4.1, it can be sampled in an open car by using the sampling device specified in 3.1. Sample from 10 to 15 locations representing cross sections from top to bottom of the car.

4.2.2 Turn the inner tube of the sampler described in 3.1 until the openings are misaligned with those of the outer tube, thus forming a closed hollow tube. Then insert into the carload, bin, or pile of dry bulk material. When in place rotate the inner tube so that the inner and outer tube openings are aligned, thus permitting the inner tube to be filled with material immediately adjacent to the tube opening. Tap or shake the sampler to facilitate filling the inner tube. Rotate the inner tube to close the sampler before withdrawing.

4.2.3 Proceed to 4.5.

**Table 1.** Units to be sampled according to lot size

| <i>Lot size, units</i> | <i>Number of units to be sampled</i> |
|------------------------|--------------------------------------|
| 110 or less            | 3                                    |
| 111 - 180              | 4                                    |
| 181 - 300              | 5                                    |
| 301 - 500              | 7                                    |
| 501 - 800              | 10                                   |
| 801 - 1,300            | 15                                   |
| 1,301 - 3,200          | 25                                   |
| 3,201 - 8,000          | 30                                   |
| 8,001 - 22,000         | 35                                   |
| 22,001 and up          | 40                                   |

4.3 *For dry bag, drum, or packaged shipments*

4.3.1 Sample from the shipment or lot at random. Base the number of units to be sampled on the size of the shipment or on the lot size as given in Table 1. A lot is defined as the particular units filled from a single batch of material and so identified by the supplier. If thus identifiable, obtain a separate sample for each lot.

4.3.2 Introduce the sampler described in 3.2 into the packaged material, rotate the sampler 360° to “core” the material and withdraw the sampler and material.

4.3.3 Proceed to 4.5.

4.4 *For shipments in slurry form*

4.4.1 If the material is well mixed, sample from two or three places at random.

4.4.2 If the mixture is suspected not to be uniform, or if for a referee test, sample an additional number of places representing all parts of the shipment. This is best done during unloading or from the receiving tank after the entire slurry shipment has been unloaded and allowed to mix thoroughly. Unless receiving tank is cleaned between each shipment, then it may be a source of contamination.

4.4.3 The sampler described in 3.3 may be used to sample the contents of tank cars prior to unloading if desired. If sampling is from a well-mixed tank, then it is recommended that the bottle be attached to a rope and not to a rod as the rod can be as long as 30 to 50 feet making it unwieldy to use. If one is sampling from a well-mixed tank then a sample can be obtained by using a T-connector or by opening a small sample outlet valve installed on the tank.

4.4.4 Proceed to 4.6.

4.5 Mix, quarter, and reduce the gross sample to the desired size for the laboratory sample, in accordance with the procedure given in TAPPI T 605 “Reducing Gross Samples of Granular or Aggregate Material to Testing Size” or by other equally effective means, and store in a clean container.

4.6 Thoroughly mix together the portions of the liquid sample. Fill a clean container with the desired quantity of the mixture to comprise the laboratory sample for testing.

## 5. Safety precautions

All prevailing local safety rules should be followed while sampling products from tanks or bins.

## 6. Report

Reference this sampling procedure in the report of the specific test method used.

## 7. Precision

A precision statement is not applicable for this Standard Practice.

## 8. Keywords

Pigment, Fillers, Sampling.

## 9. Additional information

9.1 Effective date of issue: To Be Assigned.

9.2 Editorially revised to make language agree with that of TAPPI T 605 and “TAPPI Test Methods and Standard Practices Regulations and Style Guidelines.” Also reclassified in 1998 as a Standard Practice since this procedure does not produce results. Changes in the 2012 version were strictly editorial.

9.3 Sampling inaccuracies may result should there be excessive stratification within bulk shipments or should bags be filled from more than one batch of material.

9.4 Related method: ASTM C322 “Sampling Ceramic Whiteware Clays.” Additional information on the determination of the number of specific samples per lot can be found in ANSI/ASQ Z 1.4.

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

