

# T 602 cm-06

TENTATIVE STANDARD – 1931  
OFFICIAL STANDARD – 1933  
REVISED – 1973  
OFFICIAL TEST METHOD – 1983  
REVISED – 1984  
REVISED – 1989  
CLASSICAL METHOD – 1997  
REAFFIRMED – 2006  
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## 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.

## Analysis of sulfuric acid

### 1. Scope

1.1 This method describes analytical procedures for the testing of sulfuric acid used in pulp and paper manufacture to determine total acidity, Baumé gravity, nonvolatile matter, iron, sulfur dioxide, and arsenic.

1.2 Elaboration on the significance of these procedures, except as shown below, is not within the scope of this method.

### 2. Significance

2.1 The use of sulfuric acid includes its application in the manufacture of chlorine dioxide for pulp bleaching, the manufacture of tall oil, and for pH control in various processes.

2.2 The value of these analytical procedures is related to production efficiency, quality of product, safety of production personnel, and health of users.

### 3. Safety precautions

3.1 Sulfuric acid is a strong corrosive acid and is dangerous if improperly handled. Avoid any skin contact. *Analyst must wear goggles with side shields at all times and wear long sleeve rubber gloves and splash apron when sampling or pouring.* If acid is spilled on the skin, wash immediately with large amounts of water. If splashed into the eyes, wash the eyes with water for at least 15 min. Get immediate medical attention.

3.2 *Clean all spills immediately.* Refer to Material Safety Data Sheet for proper cleanup methods, and disposal of spilled material.

### 4. Sampling

4.1 Sampling of sulfuric acid is not within the scope of this method. For general guidance in sampling, consult ASTM E 300 “Recommended Practice for Sampling Industrial Chemicals.”

Approved by the Standard Specific Interest Group for this Test Method



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4.2 The sample to be analyzed is considered to be that sample in a single bottle submitted to the analytical laboratory.

4.3 The size of the sample must be sufficient to perform all analyses without the reuse of any portion of the sample. A 1000-mL sample should be sufficient.

## 5. Procedure for total acidity

5.1 *Scope.* This procedure covers the determination of the total acidity of 75% to 99% sulfuric acid. Three methods are given for weighing the specimens, namely, the Dely tube, the snake tube, and the weighing bottle methods.

5.2 *Summary of procedure.* A weighed specimen of acid is diluted in water and titrated with standardized 0.5M sodium hydroxide solution using phenolphthalein as the indicator.

5.3 *Interferences.* Acids other than sulfuric and compounds that consume sodium hydroxide will affect the accuracy of this method.

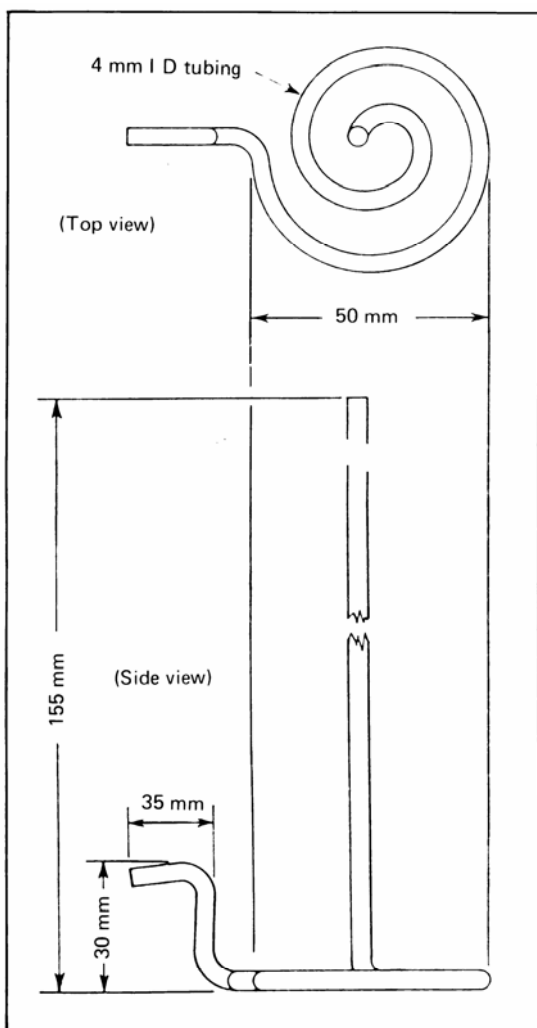


Fig. 1 Dely Tube

### 5.4 Apparatus

5.4.1 Dely tube (Fig. 1) or snake tube (Fig. 2)<sup>1</sup>, or glass weighing bottle

<sup>1</sup> Names of suppliers of testing equipment and materials for this method may be found on the Test Equipment Suppliers list in the set of TAPPI Test Methods, or may be available from the TAPPI Technical Services Department.

- 5.4.2 Buret, 100-mL.
- 5.5 *Reagents*
- 5.5.1 *Phenolphthalein indicator*, alcoholic solution (10 g per 1000 mL).
- 5.5.2 *Sodium hydroxide*, standard solution, 0.5M NaOH (see TAPPI T 610 "Preparation of Indicators and Standard Solutions").
- 5.5.3 *Water* - Reagent Grade (ASTM Type I or II) water should be used to dilute the acid.
- 5.6 *Test specimen*. Table 1 shows the suggested specimen size range for a given acid strength.

**Table 1.** Specimen size for total acidity

| <i>H<sub>2</sub>SO<sub>4</sub></i> , % | <i>Specimen size, g</i> |
|----------------------------------------|-------------------------|
| 98                                     | 1.9 to 2.2              |
| 94                                     | 2.0 to 2.3              |
| 90                                     | 2.1 to 2.4              |
| 85                                     | 2.2 to 2.6              |
| 80                                     | 2.3 to 2.7              |
| 77                                     | 2.4 to 2.8              |
| 75                                     | 2.5 to 2.9              |

### 5.7 *Test procedure*

5.7.1 *Dely tube method*. Invert the sample bottle several times. (Hold stopper in tight.) Insert the long arm of a dry, weighed Dely tube and withdraw by suction a convenient size specimen depending on the acid strength as given in Table 1. Invert the Dely tube and wipe the acid from the long arm with disposable tissue. Reweigh to the nearest 0.001 g and record the weight of the specimen. Incline the tube so that the acid runs back nearly to the bend of the short arm. Attach the short arm to an elevated water reservoir by means of a rubber tube closed near the glass beaker containing approximately 100 mL of water. Open the pinch clamp and flush the specimen into the beaker. Continue the flow of water until all acid is washed from the Dely tube.

**NOTE 1:** The Dely tube can be marked at points equivalent to weights given in Table 1.

**NOTE 2:** The presence of acid in the Dely tube may be detected by coloring the water in the reservoir with phenolphthalein indicator and the minimum amount of dilute NaOH solution that will produce a slight pink. The water flowing through the tube is decolorized as long as acid is present, and the appearance of a pink color indicates the absence of acid.

**NOTE 3:** The acid and water are separated by a bubble of air. This prevents the acid and water from mixing within the confines of the tube (the reaction of water plus acid causes heat - this should not occur in the tube).

Wash the long end of the Dely tube, collecting the washings in the beaker. Add 3 to 5 drops of phenolphthalein indicator solution. Record the temperature of the 0.5M NaOH solution, and then titrate the specimen to a pink end point. Record the titration to the nearest 0.02 mL.

5.7.2 *Snake tube method*. Invert the sample bottle several times. (Hold stopper in tight.) Insert the capillary end of a dry, weighed snake tube and withdraw by suction a convenient size specimen depending upon the acid strength as given in Table 1. Invert the tube so that the double bend is in a horizontal position. Wipe the acid from the capillary with disposable tissue. Reweigh to the nearest 0.001 g and record the weight of the specimen. Submerge the capillary of the tube in approximately 100 mL of water contained in the 400-mL beaker. Force the weighed specimen from the tube by a stream of water from a wash bottle by placing the delivery tip in the exposed end of the snake tube. Wash the tube with 50 to 70 mL of water. Remove the tube and wash the inside and outside free of acid. Swirl the contents of the beaker gently while washing. Accumulate all washings in the beaker and add 3 to 5 drops of phenolphthalein indicator solution. Record the temperature of the 0.5M NaOH solution, and then titrate the specimen to a pink end point. Record the titration to the nearest 0.02 mL.

**NOTE 4:** Do not introduce the water into the snake tube too rapidly, as this will cause spattering.

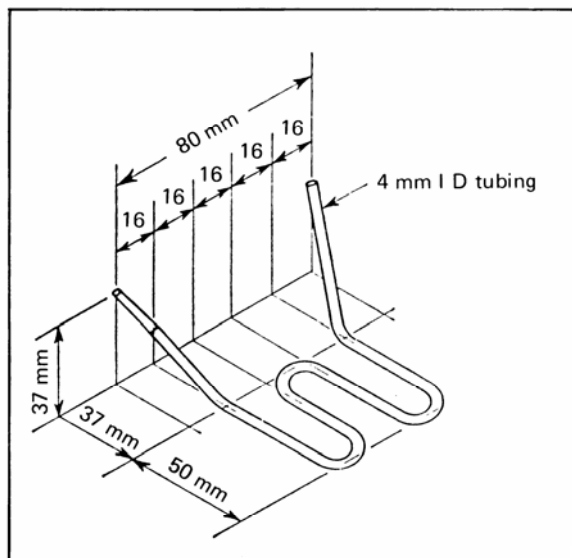


Fig. 2 Snake tube

5.7.3 *The weighing bottle method.* Weigh a dry stoppered weighing bottle and introduce a convenient size specimen depending upon the acid strength as given in Table 1. Reweigh to the nearest 0.001 g and transfer the specimen to a 400-mL beaker. Approximately 20 mLs of water should be in the beaker. Rinse the weighing bottle well with water, mixing well after the acid sample is first added, into the beaker to a total volume of 150 to 170 mL. Add 3 to 5 drops phenolphthalein indicator solution. Record the temperature of the 0.5M NaOH solution and then titrate the sample to a pink end point. Record the titration to the nearest 0.02 mL.

#### 5.8 Calculations

5.8.1 Correct the buret reading for calibration errors and record as  $V$  the corrected delivered volume at the recorded temperature.

5.8.2 Calculate the total acidity as percentage of sulfuric acid as follows:

$$\text{Sulfuric acid, \%} = \frac{VN \times 0.04904 \times 100}{W}$$

where:

$V$  = NaOH solution required for titration of the specimen, corrected mL  
 $N$  = normality of the NaOH solution  
 $W$  = amount of specimen used, g

5.9 *Report.* Report as a test result the average of duplicate determinations in percentage of sulfuric acid to the nearest 0.01%.

#### 5.10 Precision

5.10.1 Repeatability (within a laboratory) = 0.29% abs.

5.10.2 Reproducibility (between laboratories) = 0.39% abs.; in accordance with the definition of these terms in TAPPI T 1200 "Interlaboratory Evaluation of Test Methods to Determine TAPPI Repeatability and Reproducibility."

5.10.3 These values are based on interlaboratory study on three samples containing 80%, 90%, and 95% sulfuric acid. Duplicate determinations were performed by one analyst in each of ten laboratories on alternate days.

## 6. Baumé gravity

6.1 *Scope.* This procedure describes the determination of the Baumé gravity of concentrated sulfuric acid by means of a glass hydrometer in the range from 57° to 66.3° Baumé. The Baumé gravity is determined at 15.5°C (60°F).