

T 418 cm-07

OFFICIAL STANDARD – 1961
SUGGESTED METHOD – 1972
OFFICIAL TEST METHOD – 1980
REVISED – 1985
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Organic nitrogen in paper and paperboard

1. Scope

1.1 This method is used for the determination of nitrogen from glue, casein, urea- and melamine-formaldehyde resins, amine and amide polymers, and other nitrogenous organic materials in paper and paperboard.

1.2 The determination does not include all the nitrogen in nitro compounds, nitrates, nitrites, azo, hydrazine, cyanide, or pyridine ring-type compounds, which are not normally found in paper and paperboard.

1.3 Further information about alternate determination of nitrogen compounds such as the Hengar technique can be found in the literature (1).

2. Summary

2.1 This method is a modification of the Kjeldahl procedure, in which compounds that yield their nitrogen as ammonia are digested with concentrated sulfuric acid, using potassium sulfate to raise the boiling point and mercuric oxide as a catalyst.

2.2 The organic matter is decomposed and the nitrogen is fixed as ammonium sulfate in the excess acid. The solution is made alkaline. The ammonia is distilled off and determined by titration with a standard acid.

3. Significance

3.1 This method is useful in estimating the amount of the usual nitrogenous additives present in paper.

3.2 To convert nitrogen content to additive content, the nitrogen content of the additive must be known or determined. Such information may be available in the literature or from the supplier.

3.3 To estimate the amount of a specific additive present, it must be known that other nitrogenous compounds are absent or the amount of nitrogen from other sources must be known.

4. Apparatus

- 4.1 *Kjeldahl apparatus*, with 500-mL or 800-mL flasks and a digestion rack.
- 4.2 *Bulb or scrubber type trap*, connected to the flask with a rubber stopper and to the water-cooled condenser with rubber tubing. The condenser tube is made of borosilicate glass or block tin and the discharge end is connected to a bent glass delivery tube, the lower end of which is drawn out to a bore of about 3 mm.
- 4.3 *Automatic potentiometric titrator* (optional).
- 4.4 *Other apparatus*: Erlenmeyer flask, 500-mL; graduated cylinder, 100-mL; buret, 50-mL; glass beads, mossy zinc, or boiling chips for the flask to prevent bumping; powder funnel.
- 4.5 Semiautomated or automated Kjeldahl system (optional), such systems may provide automated digestion, distillation, or both.

NOTE 1: Premixed “tablets” of K_2SO_4 and HgO are commercially available. They may be used, if desired. Follow manufacturers instructions, using approximate amount of material shown in Section 9.1. Other alternative catalysts that do not contain HgO have not been shown to provide equivalent results and should not be used.

5. Reagents

- 5.1 *Potassium sulfate*, anhydrous K_2SO_4 .
- 5.2 *Mercuric oxide*, HgO .
- 5.3 *Sulfuric acid*, concentrated H_2SO_4 (sp gr 1.84).
- 5.4 *Sulfuric or hydrochloric acid*, 0.1N, standardized in accordance with TAPPI T 610 “Preparation of Indicators and Standard Solutions.”
- 5.5 *Sodium hydroxide solution*, approximately 50% by weight. Dissolve 1030 g NaOH in 1000 mL of water, or use a 50% solution as supplied by manufacturer.
- 5.6 *Sodium thiosulfate solution*. Dissolve 80 g of $Na_2S_2O_3 \cdot 5H_2O$ in 1000 mL of water.
- 5.7 *Boric acid-indicator solution*. Dissolve 43 g H_3BO_3 in water, then add 6 mL of methyl red indicator and 4 mL of methylene blue indicator (each 0.1 g in 100 mL of 95% ethyl alcohol). Dilute to 100 mL with distilled water. Keep the solution in a borosilicate glass bottle. It is stable for at least five months.
- 5.8 *Sucrose or dextrose*, for blank determination.

6. Safety precautions

- 6.1 Concentrated sulfuric acid and strong alkali solution used in the procedure are hazardous and must be handled with proper care. A shield of safety glass or plastic must be placed in front of the digestion and distillation apparatus.
- 6.2 Mixing of sulfuric acid with water (9.2) and with alkali solution (9.5) must be performed carefully and cautiously. A face shield and protective gloves must be worn for protection in the event of a violent reaction or glassware breakage.
- 6.3 Mercuric oxide used in the procedure is poisonous and must be handled with care. Any contact of it with skin or by inhalation must be avoided.
- 6.4 The residue remaining in the flask after digestion and distillation contains mercury which will contribute to environmental pollution if discarded carelessly. The residue must be filtered off and collected for reprocessing or disposal in accordance with all applicable state and federal waste disposal regulations.

7. Sampling

Obtain a sample of paper or paperboard in accordance with TAPPI T 400 “Sampling and Accepting a Single Lot of Paper, Paperboard, Containerboard, or Related Product.”

8. Test specimens

Condition the sample in the atmosphere near the balance to the moisture equilibrium. Cut strips or pieces about 1 cm^2 in area and weigh two 2-g test specimens to the nearest 0.001 g. At the same time, weigh specimens for the determination of moisture in accordance with TAPPI T 412 “Moisture in Paper and Paperboard.”