WI 220808.03

T 622

BALLOT NO. 03 SARG

DRAFT NO. 01

DATE October 26, 2023

WORKING GROUP CHAIR Walter Rantanen

SUBJECT CATEGORY Chemical Properties

RELATED METHODS See "Additional Information"

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 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 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 sodium hydrosulfite

(Proposed Confirmation of Classical Method T 622 cm-10)

1. Scope

The method describes the procedure for determining the percentage of active chemicals in sodium hydrosulfite ($Na_2S_2O_4$) products.

2. Summary

This procedure is based on coupling sodium hydrosulfite with formaldehyde to form a stable sodium formaldehyde sulfoxylate, which is titrated with iodine. At the same time, any sulfites present are rendered unreactive

to iodine by the formaldehyde. From the titration of the sulfoxylate with iodine, the percentage of $Na_2S_2O_4$ in the sample is calculated.

3. Significance

3.1 This procedure determines the percentage of active chemicals in commercial sodium hydrosulfite.

3.2 The results obtained measure the active chemicals present at the point where the formaldehyde reagent is added to the sample. Since sodium hydrosulfite is an active reducing agent, exposure to humid air from the time the lot is sampled until it is analyzed could seriously alter the strength of active chemicals.

3.3 Sulfide and thiosulfate anions interfere with the procedure and can lead to erroneous results. Commercial sodium hydrosulfite contains significant amounts of thiosulfate; however, no specific recommendations to correct for this interference are available.

4. Apparatus

4.1 *pH meter,* direct-reading pH meter with a glass electrode and calomel reference electrode.

4.2 *Weighing bottle,* 25 mm × 40 mm, equipped with stopper.

4.3 *Sample bottles,* 1 L, tightly-stoppered, dark colored.

4.4 *Other apparatus:* 25-mL and 100-mL graduated cylinders, 10-mL pipet, 500-mL volumetric flask, 50-mL burets, 250-mL beakers, powder funnel, 75-mm-long stem funnel, sample thief, wide-mouthed jars or bottles, spatula.

5. Reagents

5.1 *Acetic acid solution,* 20% solution of glacial acetic acid.

5.2 *Formaldehyde-sodium carbonate solution.* Mix 35 mL of fresh, clear 37% formaldehyde solution with 50 mL of distilled water and adjust the pH to between 8 and 9 with approximately 0.3 g Na_2CO_3.

NOTE 1: If numerous assays are to be made, several liters of the pH-adjusted formaldehyde solution may be prepared and kept in dark bottles at a temperature above 18°C to avoid polymerization.

5.3 *Iodine,* 0.1*N* iodine, accurately standardized (see TAPPI T 610 “Preparation of Indicators and Analytical Reagents, and Standardization of Volumetric Solutions”).

NOTE 2: If numerous assays are to be made, it is convenient to prepare several liters of iodine solution. Keep the solutions in tightly stoppered, dark colored bottles at a constant temperature below 27°C.

5.4 *Sodium thiosulfate,* 0.1*N* sodium thiosulfate, accurately standardized (see T 610).

5.5 *Starch indicator,* 1% starch solution (see T 610).

6. Safety precautions

6.1 Sodium hydrosulfite undergoes thermal decomposition at 55°C. If heated rapidly, it can readily explode. The decomposition process results in the evolution of toxic fumes of SO_2.

6.2 Sodium hydrosulfite will react with moisture, steam, or acids to produce toxic and corrosive materials. This reaction can take place with body moisture to cause skin irritation. When sodium hydrosulfite is handled, protective equipment should be used.

6.3 Inhalation or ingestion can cause tissue burns, irritation, and burns by liberation of sulfurous acid.

6.4 The Occupation and Safety Administration (OSHA) has determined that formaldehyde is a possible cancer hazard based on animal data. OSHA Standard 29 CFR 1910.1048 should be consulted for specific details regarding respiratory, skin and eye protection against formaldehyde. The standard also provides for establishing regulated areas, respiratory protection programs, housekeeping, medical surveillance, record keeping, and employee information and training when necessary.

7. Sampling

7.1 *Drums.* Use a suitable sampling thief which extends to the bottom of the drum and collects material along its length. Take three such samples from different locations in the drum and place in a dry, 1-L sample bottle. Tightly stopper the sample bottle and roll it until mixing is complete.

7.2 *Lots or shipments.* Use the sample thief described above to remove one sample from each drum. Place each sample in a separate dry, small, wide-mouthed jar.

8. Test specimens

From each test unit of sample weigh triplicate specimens for analysis if dealing with the contents of a drum. If the analysis is to determine the variability of a lot or shipment, single weighing of each specimen is used.

9. Procedure

9.1 Fill a weighing bottle 3/4 full of sample and immediately stopper. Take care to avoid unnecessary exposure of the sample to air. Weigh the sample and container to the nearest 0.1 mg.

9.2 Transfer 85 mL of the formaldehyde reagent to a 500-mL volumetric flask, using a long stem funnel taking care not to wet the inside neck of the flask. Pour the contents of the weighing bottle into the volumetric flask using the powder funnel while gently swirling the flask. Rinse the funnel and neck of the flask with 5 mL of formaldehyde solution. Swirl the flask until all of the sample is dissolved.

9.3 Immediately after transferring the sample from the weighing bottle, stopper it. After dissolution of the sample is complete, weigh the empty sample bottle to determine the net weight of the sample analyzed. After the

hydrosulfite has dissolved, wash the funnel and neck of the flask with freshly boiled and cooled distilled water, fill the flask to the mark, and mix.

9.4 Pipet 10 mL of this sulfoxylate solution into a 250-mL beaker, add 90 mL of freshly boiled and cooled distilled water and 5 mL of 20% acetic acid.

9.5 Titrate with 0.1*N* iodine, adding the starch indicator near the end point. Add iodine until a deep blue color persists, then 0.5 mL in excess. Back titrate the solution with 0.1*N* sodium thiosulfate until the blue color just disappears.

10. Calculations

$$\% \ Na_2S_2O_4 = \frac{(C_1N_1 - C_2N_2) \times 0.0435 \times V_1 \times 100}{(W_1 - W_2) \times V_2}$$

where

C_1 = volume of iodine titrant

N_1 = normality of iodine

C_2 = volume of sodium thiosulfate titrant

N_2 = normality of thiosulfate

V_2 = volume of sulfoxylate solution titrated, 10 mL

V_1 = total volume of sulfoxylate solution, 500 mL

W_1 = weight of sample and weighing bottle, g

W_2 = weight of weighing bottle, g

11. Report

11.1 Report the average of three determinations to the nearest 0.1 weight percent as the assay of the drum.

11.2 For lot or shipment variability, each value is reported to 0.1 weight percent.

12. Precision

12.1 Repeatability (within laboratory) = 0.53%.

12.2 Reproducibility = not known.

12.3 Comparability = not known.

12.4 The precision statement is in accordance with the definitions of these terms in TAPPI T 1200 "Interlaboratory Evaluation of Test Methods to Determine TAPPI Repeatability and Reproducibility" and is based on data from one laboratory analyzing 94% active material.

12.5 The 2010 edition added section 6.4 (OSHA information).

13. Keywords

Sodium hydrosulfite

14. Additional information

14.1 Effective date of issue: To be assigned.

14.2 Chemical reactions:

$$Na_2S_2O_4 + 2HCHO + H_2O \rightarrow NaHSO_2 \bullet HCHO + NaHSO_3 \bullet HCHO$$

$$NaHSO_2 \bullet HCHO + 2I_2 + 2H_2O \rightarrow NaHSO_4 + HCHO + 4HI$$

14.3 Sodium hydrosulfite, $Na_2S_2O_4$ is also known as sodium sulfoxylate or sodium dithionite. The name "sodium hydrosulfite" is applied also to $NaHSO_2$. Still more confusion results when "sodium hyposulfite" is applied to the compound $Na_2S_2O_4$.

14.4 This revision comprises essentially the same general procedure as in former editions and contains editorial changes. To improve end point detection, the last revision prescribed adding excess iodine and back titrating with sodium thiosulfate. In addition, the sample weighing procedure was changed to a different technique to minimize sample oxidation and to allow for the convenient addition of the sample to the formaldehyde reagent. This latter change is reported to give more consistent results because it avoids sample decomposition through "hot spots" and poor mixing.

14.5 This method, formerly T 622 os-78, has been reclassified as a Classical Method. Such procedures are no longer in common use or have been superceded by advanced technology; they are technically sound, have a history of use, and contain a body of literature references that make their preservation valuable. In 2010, a section on OSHA requirements was added

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