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T _____ 628 _____

BALLOT NO. _____ 3 SARG _____

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DATE _____ 06/01/2023 _____

WORKING GROUP
CHAIR _____ To Be Determined _____

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 rosin size

Proposed RECONFIRMATION of Classical Method T 628 cm-10

1. Scope and significance

1.1 This method deals with the analysis of those rosin sizes made ordinarily by reacting rosin with an alkali. Separate procedures are given for paste size, dry size, and size emulsions. The procedures do not apply to the analysis of colloiddally protected sizes or those containing added ingredients, such as waxes or oils.

1.2 It is usually desirable to express results as percentages of the original material and also of the total solids, i.e., on the "dry basis."

2. Apparatus

2.1 *Electric drying oven*, thermostatically controlled to maintain a temperature of $105 \pm 1^\circ\text{C}$. It is advisable also to have under the lower shelf an auxiliary heating coil controlled by a separate switch (in series with a rheostat, if necessary) adjusted so that starting from a temperature of 105°C , an oven temperature of 112° to 115°C is attained in 15 min after the auxiliary heater is turned on.

2.2 *Flat glass or metal weighing dishes*, diameter 50 mm, depth 12 mm (aluminum is preferable).

2.3 *Light filter* (optional). This is required for the determination of free rosin in paste size by the titration procedure only and is a combination of a light green gelatin filter, such as a Wratten No. 52 (in "C" glass), and an IR absorbing glass filter with a transmission range of 3000 \AA to 7000 \AA and a peak transmission of about 60% at 5000 \AA . These filters are mounted in a box so that the IR absorbing filter is closest to the light source in the box and the bottom of the filter combination is about 51 mm (2 in.) above the top of a 60-W frosted tungsten electric bulb. The IR absorbing filter serves to absorb the heat emitted by the light source and thus protects the green filter.

3. Reagents

3.1 *Acid-free ether*. Wash a quantity of diethyl ether with sodium carbonate solution and then several times with water. Test the prepared ether with a moist piece of sensitive blue litmus paper, which shall not change color when completely submerged in it for 15 min.

NOTE 1: The presence of peroxides in ether constitutes a safety hazard, as serious explosions have resulted from laboratory work with ether containing peroxides. Test for their presence as follows: Mix 5 mL of an aqueous solution of benzidine (Eastman Kodak No. 33), saturated in the cold, with 5 mL of a saturated NaCl solution. Add a few drops of very dilute, freshly prepared ferrous sulfate solution (a particle of FeSO_4 the size of a pinpoint dissolved in 5 mL of water). Then add 3 to 5 drops of ether to this mixture. The development of a clear blue coloration after a few minutes indicates the presence of peroxides.

3.2 *Neutral alcohol*. If necessary, neutralize 95% (by volume) ethyl alcohol with a small amount of alcoholic KOH. Then test with a piece of sensitive blue litmus paper, which shall not change color when completely submerged for 15 min.

3.3 *Alcoholic KOH solution*, approximately 0.5N. Dissolve 33 g of KOH in 1 L of 95% (by volume) ethyl alcohol. Filter off any insoluble carbonates and store in a bottle protected from CO_2 of the air by means of tubes containing soda lime.

3.4 *Alcoholic KOH solution*, 0.1N. Pipet 100 mL of the 0.5N alcoholic KOH into a 500-mL volumetric flask and dilute to 500 mL with neutral ethyl alcohol. Standardize against 0.1N HCL, using phenolphthalein indicator.

3.5 *Hydrochloric acid*, 0.1N, accurately standardized.

3.6 *Hydrochloric acid*, 0.5N, accurately standardized.

3.7 *Methyl orange indicator solution*, 1 g/L.

3.8 *Phenolphthalein indicator solution.* Dissolve 5 g of phenolphthalein in 500 mL of 50% alcohol. Neutralize by adding very dilute alkali cautiously until a faint pink color appears, then just remove the color with a drop or two of approximately 0.01N acid.

3.9 *Anhydrous isopropyl alcohol,* neutral to moist, sensitive litmus paper.

3.10 *Isopropyl alcoholic KOH solution,* approximately 0.5N. Dissolve 33 g of KOH in 1 L of anhydrous isopropyl alcohol. Filter off any insoluble carbonates and store in a bottle protected from CO₂ of the air by means of tubes containing soda lime. Standardize against 0.5N HCl, using thymol blue indicator.

3.11 *Thymol blue indicator solution,* 1%.

NOTE 2: The last two reagents are required only for the determination of free rosin by the titration method.

4. Paste size

4.1 Rosin size in paste form normally contains 50 to 80% solids. Frequently water will partially separate out on standing and care is required to obtain a representative sample.

4.2 *Sampling*

4.2.1 If the material is supplied in drums, heat the drums until the size is melted, guarding against dilution and loss of moisture. Agitate thoroughly and withdraw approximately equal portions from 10% of the drums of each batch number. Thoroughly mix the samples and fill a moisture-tight quart glass jar with the composite.

4.2.2 If the material is received in a tank car, heat the car until the size is melted and collect four samples by lowering through the dome of the car a rod to which is attached a wide-mouthed bottle or other suitable container closed with a stopper to which a wire is attached, enabling the sampler to collect the sample at any designated level. Collect samples at four different levels: 1 ft (305 mm) below the surface, 1 ft (305 mm) above the bottom, and at two equally spaced levels representing the second and third fourths of the car. Thoroughly mix the samples and fill a moisture-tight quart glass jar with the composite.

4.2.3 As an alternative method, tank cars may be sampled during unloading by collecting four samples from the discharge line representative of each quarter of the car's contents.

4.3 *Preparation of sample.* Stir each laboratory sample well to insure thorough mixing. If separation of water has occurred, warm the size until with additional stirring it becomes uniform, taking care to avoid loss of water.

4.4 *Moisture and volatile matter*

4.4.1 Weigh 1 g of the original sample (to the nearest 0.5 mg) into a flat weighing dish. An aluminum dish is preferable since, because of its conductivity, the dish warms up faster, resulting in more efficient puffing and faster drying of the size. Place the dish containing the sample on the lower shelf of the oven adjusted to 105 ± 1°C. Turn on the auxiliary heater for approximately 15 min. This procedure brings about a rapid heating of the sample which causes it to puff up and dry very rapidly. The oven temperature should reach 112° to 115°C by the end of the period required to accomplish puffing of the size. After the first 15-min period, remove the dish to an upper shelf in the oven. Turn off the

auxiliary heater, which will allow the temperature to drop gradually to 105°C, and continue the drying for 30 min longer. Cool in a desiccator and weigh. Replace the dish in the oven on an upper shelf and dry for an additional 15 min. Cool and reweigh. If the second weighing shows a loss of more than 0.5 mg, repeat the drying at 105°C for 15-min periods. If the third or subsequent weighing shows an increase (due to oxidation), use the minimum weight in the calculations.

4.4.2 If the auxiliary heater is not available, dry for 1 h at 105°C and then repeat for 0.5-h intervals until constant or minimum weight is attained.

4.4.3 Calculate the percentage loss as moisture and volatile matter; also the percentage of dry solids. Duplicate determinations should check within 0.1.

4.5 *Free rosin.* Two methods are given for determining the degree of saponification of rosin. These methods are based on extraction and titration, respectively. The extraction method is more time consuming, and with this method there is a danger of hydrolysis of some of the combined rosin, giving high results. The extraction method determines free, or unneutralized, resin acids, plus all of the unsaponified esters and neutral material soluble in ether. The fraction determined by the extraction method is reported as extractable free rosin. The titration method is faster but assumes a knowledge of the acid number of the original rosin. It determines the free resin acids and by calculation includes only that portion of unsaponified esters and neutral materials originally associated with the uncombined resin acids in the finished size. The fraction determined by the titration procedure is reported as titratable free rosin.

4.5.1 *Extractable free rosin.*

4.5.1.1 Weigh approximately 5 g of the sample (to the nearest 5 mg) into a 100-mL tared beaker; dissolve by stirring in 10 mL of *neutral* ethyl alcohol, adding the alcohol slowly to prevent formation of lumps; transfer to a 250-mL separatory funnel (*A*); rinse the beaker three times with a total of 40 mL of acid-free ether and finally with several portions of water, not to exceed 35 mL total, adding the rinsings to the separatory funnel. Mix thoroughly by gentle shaking, avoiding violent agitation which might result in a difficultly separable emulsion.

4.5.1.2 Let the layers separate and draw off the lower water layer into a second separatory funnel (*B*). Extract this water layer with 25 mL of acid-free ether, draw off the water into a third separatory funnel (*C*), and add the ether extract to that in Funnel *A*. Wash the combined ether extracts in Funnel *A* with two 25-mL portions of water, adding the wash water to the solution in Funnel *C*. Transfer the ether extracts in Funnel *A* to a weighed Soxhlet flask.

4.5.1.3 Finally, extract the total wash water in Funnel *C* with 25 mL of ether, first using the ether to rinse Funnels *A* and *B*. Discard the water layer in Funnel *C*. Then wash the ether extract still contained therein with 25 mL of water. Draw off and discard the water and transfer the ether extract to the Soxhlet flask containing the main ether extract. Evaporate the ether by means of a water or steam bath; then place the flask in the oven at $105 \pm 1^\circ\text{C}$ and dry for 1 h. Cool in a desiccator and weigh. Repeat the drying for 0.5-h intervals until a constant or minimum weight is obtained. Calculate the weight of free rosin to percentage of the original sample.

NOTE 3: Rotate the flask frequently during drying to break the surface so the moisture can be liberated more rapidly. Addition of 10 mL of alcohol to the rosin aids in the drying. Long duration of heating may result in oxidation of the rosin with a consequent increase in weight.

CAUTION: Because of the explosive hazard attending the use of ether, procedures involving its use should be carried out under a hood with adequate ventilation away from open flames and exposed heating elements. Be certain that the ether is free of peroxides (see Section 3).

4.5.2 *Titrateable free rosin (1)*

4.5.2.1 This method is based on the titration in nearly monochromatic light of a rosin size solution in combination with an indicator. The light is obtained by means of a filter whose transmission band is nearly the same as the absorption band of the indicator in alkaline condition. The indicator used is thymol blue, which changes color at a pH range of 8.0 to 9.6 (titration with phenolphthalein in normal light is satisfactory with pale rosin). Thymol blue in acid condition transmits nearly the entire spectrum and thus appears colorless or a pale pink. In an alkaline condition, a strong absorption band appears, thus producing a blue color. In place of noting the usual color change, the end point is noted by observing the point at which the light intensity is sharply decreased. Complete absorption of light will not be obtained because the transmission band of the light filter and the absorption band of the indicator in alkaline condition are not identical.

4.5.2.2 Weigh 4.0 to 4.5 g of the size (to the nearest 5 mg) into a 250-mL beaker. Dissolve the size in 100 mL of neutralized anhydrous isopropyl alcohol by adding the alcohol a few drops at a time and stirring it in until a smooth semiliquid is obtained which can be diluted with larger amounts of the alcohol without the formation of lumps. Add exactly 0.5 mL of 1% thymol blue indicator solution and titrate with 0.5*N* isopropyl alcoholic KOH solution over the Wratten filter.

4.5.2.3 The color after the addition of the indicator is yellow-green. As the addition of alkali progresses, the color changes to a darker green and finally there is a sharp decrease in the amount of light transmitted through the solution. This is taken as the end point. The color range will vary, depending on the color of the rosin used in the size, but the sharp decrease in the amount of light transmitted through the solution will be apparent.

4.5.2.4 *Calculation:*

$$\text{Titrateable free rosin, \%} = \frac{\text{mL } 0.5N \text{ KOH} \times 0.173 \times 100}{\text{grams of sample}}$$

NOTE 4: The above calculation assumes a rosin acid number of 162. The factor 0.173 is obtained by dividing 28.06 (the number of milligrams of KOH in 1 mL of 0.5*N* solution) by 162. If the acid number of the rosin from which the size was made is known to be different from 162, the factor should be changed accordingly.

NOTE 5: A value approximately that determined by extraction as described in 4.5.1 may be obtained from titration results by the following calculations:

Let *A* = free resin acids, %
B = nonacids in original rosin, %
C = extractable free rosin, %

D = acid number of original rosin

then,

$$A = \frac{\text{mL } 0.5N \text{ KOH} \times 0.151 \times 100}{\text{grams of sample}}$$

$$B = \frac{(185.5 - D) \times 100}{185.5}$$

and

$$C = A + B$$

Report as extractable free rosin (determined by titration).

4.6 *Total rosin*

4.6.1 Weigh out 3 to 4 g of the size sample (to 5 mg) and transfer to a 250-mL separatory funnel with about 100 mL of distilled water. Add 2 mL of concentrated HCl, then 25 mL of diethyl ether. (This need not be acid-free.) Shake thoroughly, relieving the pressure at intervals through the stopcock. Allow the ether to separate completely and draw off the water layer into a second funnel. Wash the ether layer with three 25-mL portions of water and transfer the wash waters to the second funnel. Run the ether layer into a weighed Soxhlet flask and rinse the funnel with fresh ether, allowing it to run into the second funnel, and add 25 mL more of either. Shake as before and discard the water layer. Wash the ether layer with two 25-mL portions of water. Add the ether from the second funnel to the Soxhlet and evaporate the ether by means of a steam or hot-water bath.

4.6.2 Dry and weigh the extracted rosin in an oven at 105°C as described in 4.5.1 and calculate to percentage of the original sample.

4.7 *Combined rosin.* This is calculated as the difference between the percentages of total and free rosin.

4.8 *Unsaponifiable material.* Weigh 3 to 4 g of the sample (to 5 mg) into a 250-mL Erlenmeyer flask. Add 50 mL of approximately 0.5N alcoholic KOH and boil for 2 h under a reflux condenser. Remove from the reflux condenser and place on the steam bath. Evaporate the alcohol to a volume of approximately 15 mL. Cool and transfer to a 25-mL separatory funnel. Rinse the flask three times with a total of 40 mL of acid-free ether, and finally with several portions of CO₂-free distilled water, totalling 100 mL, adding the rinsings to the separatory funnel. Proceed as in the determination of free rosin, with one modification: before adding the ether extracts to the weighed Soxhlet flask, wash with an additional 10 mL of water. Draw off the water layer and test by adding a drop of phenolphthalein. If, as indicated by a pink color, sodium soaps are present, wash with successive 10-mL portions of water until no reaction with phenolphthalein is obtained. Finally dry the ether extract, weigh, and calculate the weight of unsaponifiable matter to percentage of the original sample.

4.9 *Ash.* Weigh approximately 1 g of the sample (to the nearest 0.5 mg) into a weighed platinum crucible. Place the crucible in a round hole cut in the center of a thick piece of asbestos board. The hole should be large enough to allow the lower half of the crucible to extend below the board. The board is used to keep the burner flame from igniting the vapors produced. Heat cautiously with a gas burner until all volatile matter has been driven off. Increase the temperature and ignite until practically all carbonaceous material is burned off. Finally, incline the crucible at about a 45° angle on a clay or fused-silica triangle, heat to a dull red color, and rotate the crucible until all the carbon has been burned away. Cool in a desiccator and weigh. Calculate the percentage of ash found.

NOTE 6: Complete combustion of the carbon can be facilitated by moistening the cooled ash with a few drops of water, drying on the steam bath and continuing the ignition. The water treatment may be repeated if necessary.

4.10 *Alkalinity of ash.* Place the crucible from the ash determination in a 250-mL beaker, cover with distilled water and boil. When the ash is dissolved, cool and titrate to a pink color with 0.1N HCl, using 2 drops of methyl orange indicator. Calculate the alkalinity in terms of sodium carbonate as a percentage of the original sample:

$$1 \text{ mL } 0.1N \text{ HCl} = 0.0053 \text{ g Na}_2\text{CO}_3$$

4.11 *Free alkali*

4.11.1 This method is based on the insolubility of sodium carbonate and sodium bicarbonate in anhydrous isopropyl alcohol in the presence of rosin size.

4.11.2 Weigh 5 to 7 g of the size (to the nearest 5 mg) into 100-mL beaker and dissolve in 35 mL of *neutral* anhydrous isopropyl alcohol by adding the alcohol a few drops at a time and stirring it in until a smooth semiliquid is produced which can be diluted further without the formation of lumps. Place 15 mL of isopropyl alcohol over the surface of the mixture in order to prevent the size solution from creeping up the sides of the beaker. Place the beaker in an airtight container to protect the contents from acid fumes and moisture absorption and let it stand overnight. Filter, using a dry piece of filter paper characterized as an ashless, acid washed paper with medium porosity, medium flow rate, and a particle retention size of 8µm for use with gravimetric analysis. and wash the residue with small portions of hot, neutral isopropyl alcohol (allowing each washing to drain completely) until the residue is free of rosin size. Remove the filtrate and place a clean 250-mL beaker or flask under the funnel. Wash the filter paper with small portions of distilled water, collecting the solution in the beaker and using in all about 100 mL of water which previously has been boiled free of CO₂ and cooled to room temperature. Add 5 to 8 drops of phenolphthalein indicator and titrate with 0.1N HCl until the pink color disappears. Record the titration (*a*). Then add 2 drops of methyl orange indicator and continue the titration until the color changes from yellow to pink. Record the total titration (*b*). From the titrations calculate the percentage of free sodium carbonate and bicarbonate:

If a = mL of acid to phenolphthalein end point, and b = mL of acid for total titration, then

$$\% \text{Na}_2\text{CO}_3 = \frac{2a \times 0.0053 \times 100}{\text{grams of sample}}$$

and

$$\% \text{NaHCO}_3 = \frac{(b - 2a) \times 0.0084 \times 100}{\text{grams of sample}}$$

5. Dry size

5.1 Dry rosin size normally contains 88 to 97% of solids. The usual physical form is a light powder or small granular particles.

5.2 Sampling

5.2.1 If a carload lot is to be sampled, select ten bags or packages representing different locations in the car. Open the bags, discard the first 6 in. (152 mm) of the dry size and collect a half-pint sample from each bag at this level, equidistant from the side walls. Place all of the samples in a clean, dry container of 1-gal (3.8-L) capacity, such as a friction-top can, which can be sealed airtight.

5.2.2 For less than carload shipments, sample five to ten bags as directed above.

5.3 *Preparation of sample.* Transfer the sample from the container to a clean, dry table top and mix thoroughly with a scoop or large spatula. Quarter, reject two of the diagonally opposite quarters and remix the remaining quarters. Repeat the quartering and transfer two opposite quarters to a 1-quart (0.95-L) mason jar. The entire operation should be carried out promptly to avoid change through contact with the air.

5.4 *Moisture and volatile matter.* Weigh 2 g of the sample (to the nearest 0.5 mg) into a flat weighing dish; dry for exactly 1 h at $105 \pm 1^\circ\text{C}$, cool in a desiccator, and weigh. Calculate the percentage loss as moisture and volatile matter. Also calculate the percentage of dry solids.

5.5 Free rosin.

5.5.1 *Extractable free rosin.* Weigh 3 g of the sample (to the nearest 1 mg) into a 100-mL beaker and form a paste by adding 10 mL of *neutral* alcohol with stirring. Transfer to a separatory funnel and complete the determination as described in 4.5.

5.5.2 *Titrateable free rosin.* Weigh 2 g of the sample (to the nearest 0.5 mg) into a 250-mL beaker; mix thoroughly with 100 mL of neutral ethyl alcohol and add 10 drops of phenolphthalein indicator. If the solution does not

change color, titrate with 0.1*N* alcoholic KOH until a reddish color is formed and then add 1 to 2 mL in excess. Record the total mL of 0.1*N* KOH added as *A*. Then titrate with 0.1*N* HCl to the disappearance of the red color. Call the mL of 0.1*N* HCl used *B*. Calculate the percentage of free rosin from the formula:

$$\text{Free rosin, \%} = \frac{(A - B) \times 0.0346 \times 100}{\text{grams of sample}}$$

NOTE 7: The above calculation assumes a rosin acid number of 162. The factor 0.346 is obtained by dividing 5.61 (the number of milligrams of KOH in 1 mL of 0.1*N* solution) by 162. If the acid number of the rosin from which the size was made is known to be different from 162, the factor should be changed accordingly.

NOTE 8: If the solution turns red when the phenolphthalein is added, free rosin is absent and the solution may be used for determination of free alkali.

NOTE 9: Dark-colored samples may be titrated by use of the Wratten filter and thymol blue indicator as described for paste size.

NOTE 10: The remarks in 4.5, regarding the applicability of the two methods, apply also to dry size.

5.6 *Total rosin.* Weigh 3 to 4 g of the sample (to the nearest 5 mg) into a 100-mL beaker, emulsify with hot water, transfer to a 500-mL separatory funnel and complete the determination as described under paste size.

5.7 *Combined rosin.* This is taken as the difference between the total and the free rosin.

5.8 *Unsaponifiable material.* Weigh 3 to 4 g of the sample (to the nearest 5 mg) into a 250-mL Erlenmeyer flask and complete the determination as described for paste size.

5.9 *Ash and alkalinity of ash.* Weigh 1.0 to 1.5 g of the sample (to the nearest 5 mg) into a platinum crucible and complete the determinations as described for paste size.

5.10 *Free alkali.* Weigh 2 g of the sample (to the nearest 1 mg) into a 250-mL beaker; mix thoroughly with 100 mL of *neutral* ethyl alcohol and add 10 drops of phenolphthalein indicator. (The solution prepared for the free rosin determination by the titration method may be used.) If the solution turns red on addition of the indicator, titrate with 0.1*N* HCl until the red color disappears. Designating the mL of 0.1*N* HCl required as *A*, calculate the percentage of free NaOH in the sample from the formula:

$$\text{Free alkali as NaOH, \%} = \frac{A \times 0.0040 \times 100}{\text{grams of sample}}$$

6. Size emulsions

6.1 Rosin-size emulsions, unless they are clear, transparent solutions, represent a difficult problem of sampling. Milky or cloudy emulsions or solutions from which rosin has precipitated are very difficult to sample and analyze. Such samples must be thoroughly agitated to obtain a uniform dispersion of any settled material. Since rosin size hydrolyzes to a considerable extent when diluted with water, determinations of free rosin and free alkali are meaningless in dilute emulsions or “rosin size milk,” such as are used in paper mills. The determinations ordinarily made are those described below.

6.2 *Specific gravity.* By means of a pipet, fill a weighed 50- or 100-mL volumetric flask to the mark with the emulsion at $20 \pm 1^\circ\text{C}$. Weigh the flask and contents to the nearest 0.1 g. Divide the weight of the emulsion in grams by the capacity of the flask in milliliters and record the specific gravity to three decimal places.

6.3. *Total solids.* Pipet 25 mL of the sample into a weighed platinum dish. Evaporate on the steam bath to dryness, then heat in the oven at $105 \pm 1^\circ\text{C}$ for 0.5 h, cool in a desiccator and weigh. Repeat the drying for 0.5-h periods until the weight is constant or begins to increase. Calculate the minimum weight to percentage of total solids:

$$\text{Total solids, \%} = \frac{\text{weight in grams}}{\text{sp. gr. of sample}} \times \frac{100}{25}$$

6.4 *Ash and total alkali.* Ignite the solids in the platinum dish and determine the ash and alkalinity as described in 4.9 and 4.10.

6.5 *Total rosin.* Pipet 100 mL of the sample into a 250-mL separatory funnel. Add 2 mL of concentrated HCl and then 25 mL of diethyl ether. Complete the extraction of the rosin as directed in Section 4, except that 25-mL portions of wash water are used.

$$\text{Total rosin, \%} = \frac{\text{grams of rosin} \times 100}{100 \times \text{sp. gr.}}$$

7. Keywords

Rosin size, Moisture content, Volatiles content, Rosin, Ash, Alkali, Solids content, Density

8. Additional information

8.1 Effective date of issue: To Be Assigned

8.2 This method, formerly T 628 ts-53, has been reclassified as a Classical Method. Such procedures are no longer in common use or have been superseded by advanced technology; they are technically sound, have a history of use, and contain a body of literature references that make their preservation valuable. Changes in the 2010 edition included revision of the light filter requirements in 2.3, and a generic description of the filter paper.

Literature cited

1. Georgi, E. A., "Analysis of Rosin Size," *Paper Trade J.* **111** (7): 37 (1940).

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