ation. The initial investment in equipment is reduced and the labor of handling a second filter press is eliminated. However, it is preferable, where these considerations are not sufficiently important, to pass the second-treatment through a second filter press (see Fig. 2). This will prevent a possibility of contamination in the feed liquor to the evaporator.

Muriatic acid, although its initial cost is higher, is preferred to sulfuric acid. The use of sulfuric acid in the treatment is objectionable in that it produces sulfates which interfere with subsequent plant operations. Feed liquor containing excess sulfates may form a hard scale inside the glycerine evaporator tubes, reducing the rate of evaporation and causing shutdowns for its removal. Sulfates also contaminate the salt recovered on evaporation of the lyes, making the salt less effective in the "graining out" operation of the soap kettles.

A combination of ferric chloride and alum in which alum is used in the proportion of ½ to ¾ pounds per thousand pounds of lye produces an efficient floc for removing impurities in the lye. The purpose of the alum is to produce a voluminous floc which will aid in the settling and filtration of the finer floc of ferric chloride. The use of alum alone as the flocculating agent will give satisfactory results in most cases. However, it must be kept in mind that its use will introduce a slight amount of sulfate which will contaminate the salt recovered on evaporation. Allowance for the proportionate amounts of alum and ferric chloride in the treatment should be made by adding the corresponding amounts of Standard Alum and Standard Ferric Chloride Solutions to the spent lye sample being tested.

As a rough guide, the following table indicates normal ranges for materials used in treatments:

TABLE II

Material	Pounds of Material Per 1,000 Pounds of Lye
Alum, $Al_2(SO_4)_3 \cdot 18H_2O$	1½ to 3 pounds
Ferric Chloride, lump, $FeCl_3 \cdot 6H_2O$	¾ to 1½ pounds
Anhydrous Ferric Chloride, $FeCl_3$	½ to 1 pound
Muriatic Acid, 22° Be, HCl	3½ to 8 pounds
Muriatic Acid, 20° Be, HCl	3½ to 9 pounds
Muriatic Acid, 18° Be, HCl	4 to 10 pounds
Sulfuric Acid, 66° Be, H_2SO_4	2 to 5 pounds
Sulfuric Acid, 60° Be, H_2SO_4	2½ to 6 pounds
Filter Aid, diatomaceous	1 to 5 pounds
Caustic Soda, Flake, 95% NaOH	½ to ¾ pound

However, the amounts of materials may vary over a larger range depending on the source of the spent lyes and on individual plant kettle operations. Lyes from poor kettle stock will require greater amounts of treatment materials.

Summary

Herein is described a method of spent soap lye purification which has the following advantages:

(1) Places the control of the product under the laboratory.

(2) Consistently yields a glycerine feed liquor of the highest possible quality.

(3) Conserves equipment, labor, and treatment materials.

(4) Uses facilities which are either present in or can be readily obtained by the soap plant.

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Routine Turbidimetric Determination of Tin in Soap

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THE addition of tin salts to soap to inhibit the development of rancidity has been patented (1). No analytical methods have been reported for the determination of tin in soap and this investigation was undertaken to provide for its estimation in the concentrations of tin to be expected in this connection, i.e., 0.01% to 0.25% tin.

A simple procedure which first suggests itself, of ashing the soap and analyzing the ash for tin, leads to immediate analytical difficulties as the tin salts are either partly reduced to the volatile metal or converted to the refractory tin oxide. In either case the tin is lost for quantitative purposes. Allen, in giving ash methods for various products, notes that the negative results of some workers when tin is known to be present is probably due to poor tin recovery from the ash (2). Results of the present investigation lead to the conclusion that the use of magnesium nitrate (3, 4) as an ash-aid provides for ready and quantitative tin recovery from the ash.

The analytical literature provides many methods for the determination of tin. Of these, the usual iodimetric and gravimetric methods require higher tin concentrations than are met with in soap analysis and almost all the colorimetric methods depend on the reducing action of the stannous ion and hence suffer from their being subject to many interferences. The present work has indicated that while known stannous chloride solutions give excellent color reactions in very low concentration, stannic chloride solutions are difficult to reduce in low concentration and give discordant, and often negative, results. It has been found that the estimation of the tin as a suspension of stannic sulfide (5) best lends itself to the immediate problem.

Procedure

Preparation of Sample: Weigh a 10 ±0.1 gram sample into an 85 mm. porcelain dish containing a short, stout glass rod. Add 10 ml. of a saturated alcoholic solution of magnesium nitrate hexahydrate [$Mg(NO_3)_2 \cdot 6H_2O$].

Place the dish and contents on a hot plate over an asbestos board and warm while stirring to homogenize the mixture. Evaporate the mixture to dryness, stirring and breaking the gas bubbles which form, to avoid loss of sample. As conditions warrant, remove the board and apply the full heat of the hot plate until the mixture is thoroughly dry. It may be necessary with some samples to apply a small Bunsen flame to obtain thorough carbonization. This operation is important and may require several hours.

Place the dish in a muffle, at not more than 600°C. and maintain at this temperature for about 2 hours. Disintegrate and mix the ash with the glass rod during this period. The ash is usually dark grey in color at this point.

Remove the dish from the muffle, allow to cool and cautiously add 25 ml. of 1:1 nitric acid. Warm the contents of the dish on the steam bath to dissolve soluble material.

Turbidimetric Estimation of Tin: Filter the contents of the dish into a 125 ml. Erlenmeyer flask marked at 100 ml. volume, washing the carbon residue twice with hot water. Neutralize the filtrate with a 40% solution of sodium hydroxide to incipient precipitation of magnesium hydroxide. Add 2 ml. of concentrated hydrochloric acid and six drops of strong bromine water. Add distilled water to bring the volume of solution to 100 ml. Pass in H_2S for ten minutes. Stopper the flask and allow to stand for about fifteen minutes.

Shake the contents of the flask to suspend the stannic sulfide precipitate and quickly fill a test tube (15 mm. × 145 mm.) with the suspension to within ¾" from the top. Stopper the tube.

A blank should be run in the same manner as the sample using the same amounts of reagents.

Prepare a standard solution of stannic chloride (6) by dissolving 1.000 grams of National Bureau of Standards tin in 100 ml. of warm, concentrated hydrochloric acid in a 400 ml. beaker. Dilute with water to about 200 ml. and add strong bromine water to a permanent yellow. Transfer the solution to a 1 liter volumetric flask and bring to volume. One ml. of this solution contains 1 mg. of stannic tin.

Prepare standards by adding from 0 mg. to 15 mg. of tin in 1 mg. increments to Erlenmeyer flasks containing 4 grams of magnesium nitrate hexahydrate and 5 ml. 1:1 nitric acid. Neutralize the solution, precipitate the tin as directed for the sample, and transfer to test tubes. If the approximate concentration of tin in the sample is known before analysis, standards need only be prepared covering the range in which the tin content is expected to fall.

Compare the sample and blank with the standards by viewing horizontally. All tubes should be shaken before comparison, to produce a uniform suspension of precipitate. The amount of tin determined turbidimetrically in the blank is subtracted from the amount of tin found in the sample to obtain the correct value. Normally the blank will be found to be approximately 1 mg. of tin.

Experimental Data

Table I presents data obtained with the method. A ten gram sample was taken for analysis in each case. The per cent tin present (Column III) represents the sum of the tin originally present in the soap (0.05% tin in the cake shave soap and 0.00% in the toilet soap) and the amount added to the analytical sample as shown in Column I.

TABLE I

Turbidimetric Determination of Tin in Soap

Sample	(Column I) Percent Tin Added	(Column II) Percent Tin Found	(Column III) Percent Tin Present
Cake Shave Soap	0.00	0.04	0.05
Cake Shave Soap	0.00	0.04	0.05
Cake Shave Soap	0.05	0.09	0.10
Cake Shave Soap	0.10	0.13[1]	0.15
Cake Shave Soap	0.15	0.23[1]	0.20
Toilet Soap	0.00	0.00	0.00
Toilet Soap	0.01	0.01	0.01
Toilet Soap	0.02	0.03	0.02
Toilet Soap	0.04	0.05	0.04
Toilet Soap	0.10	0.14	0.10
Toilet Soap	0.05[2]	0.00[4]	0.05
Toilet Soap	0.08[3]	0.00[5]	0.08

[1] 50 ml. aliquot taken.
[2] No alcoholic magnesium nitrate added.
[3] 0.10% SnO_2 added.
[4] Less than 0.01%. Qualitatively present.
[5] Less than 0.01%. Qualitatively present.

Discussion

THE method assumes the absence of metals, except tin, of the hydrogen sulfide group. If tin is present in concentrations greater than 0.15% a smaller sample than recommended under procedure should be taken for analysis, or better, an aliquot of the final solution may be employed. For example, with a 10 gram sample containing 0.25% tin take a 50 ml. aliquot from the 100 ml. in the Erlenmeyer flask, add 1 ml. of concentrated hydrochloric acid, dilute to 100 ml., pass in H_2S and proceed as directed under Procedure.

Low temperature ashing is essential to avoid loss of tin. If the mass is not thoroughly charred before insertion in the muffle it will take fire with resulting danger of loss of tin.

The blank will usually approximate 1 mg. If there is question whether the turbidity is due to sulfur suspension or precipitation of tin, allow the tubes to stand overnight. A 1 mg. tin sample will settle as a precipitate on standing, whereas any turbidity due to sulfur will persist. Experience with the character of the tin precipitate will soon enable one to differentiate it at sight from the turbidity due to the reagents.

The result in Table I subscripted (4) indicates that the tin is almost totally lost during ashing if no magnesium nitrate hexahydrate is added before ignition. The result subscripted (5) shows that any tin present in the sample as SnO_2 will not be determined. Since SnO_2 is inactive as an inhibitor of rancidity it is unlikely to be added to soap, particularly since it would be economically unjustifiable as an inert ingredient.

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