HIS report covers the activities of the Committee for the years 1938-39. No official recommendations were made in 1938 and the report was postponed until the cooperative studies that had been undertaken were completed.

The following subjects have been studied by the Committee:

1. McNicoll method for rosin determination.

Under subject No. 1 above, the tabulated results have been distributed to the Committee and are published as a part of this report. The vote of the Committee was unanimously in favor of adopting the McNicoll method as a tentative procedure. It was decided to retain the present official Wolff method as an alternate procedure until such time as the members have accumulated further experience with both methods.

Referring specifically to the details of the McNicoll method as distributed to the Committee members (November 21, 1938), a few minor changes were approved, as follows:

a. Drying time and temperature in preparation of fatty acids changed to "45 to 60 min. at 105° C."

b. Rosin soda soap factor to be included, namely, 1eq NaOH = 0.368 g. rosins.

c. Change "free" fatty acid soap to "true" fatty acid soap.

d. Use 25 ml. pipette instead of 20 ml. pipette.

e. Method for preparation of fatty acids to be the same as for titer, acid and iodine number (see detail method published with this report).

f. McNicoll method gives results approximately 1% high, consequently it was approved to deduct 1% from final result. Qualitative test for rosin to be retained in this method as in the Wolff procedure.

CO₂ DETERMINATION Results of the cooperative work on the proposed Hitchcock-Divine method are published with this report. Considerable difficulties were reported by several members of the Committee and the results were, in general, not in good agreement. However, it was felt that the method holds possibilities and it was agreed to appoint a subcommittee to work out and recommend further modification.

IODINE NUMBER In addition to the above, the Committee voted to include a method for the determination of iodine number of fatty acids, the procedure following essentially that of the A.O.C.S.-A.C.S. Fat Analysis Committee on fats and oils. Preparation of fatty acids for the determination will be revised to make the procedure conform with that of preparation of fatty acids for titer and acid number. The detailed methods for all three determinations are published as a part of this report.

MATTER VOLATILE AT 105° C. (Oven Method) specifies drying to "constant weight." It was agreed to define this as follows: "Constant weight is attained when successive heating for one hour periods shows a loss (or gain) of not more than 0.1%.

The following new subjects were brought up for discussion:

FREE ALKALI A communication from a laboratory which conducts many analyses of soaps called attention to this determination and stated that errors are introduced when analyzing soaps which contain little or no alkaline builders, if the procedure of filtering the alcoholic soap solution is followed. The communicant recommended omitting filtration and titrating the hot alcoholic soap solution direct.

The Committee felt that such procedure was permissible provided no builders such as phosphates, silicates, borates, etc. were present. However, since all commercially neutral soaps contain some carbonates, it was decided that filtration could be omitted if the carbonates (as Na₂CO₃) do not exceed 0.5%. The method will be amended as follows: "Note: In testing soaps known to contain little or no alkaline salts, it is unnecessary to filter the hot alcoholic soap solution as described above. However, the filtration should be carried out in all cases where alkaline salts such as silicates, phosphates, borates, etc. are present, since these are known to affect the free alkali determination. The presence of carbonates up to 0.5% as Na₂CO₃ does not appreciably affect this determination and filtration may be omitted if the carbonate determination on a separate sample is not in excess of this figure."

SODIUM PYROPHOSPHATE In view of the extensive use of this material in certain types of soap, the Committee agreed to take up the study of methods of analysis. Samples of soap containing known amounts of this material will be made up and distributed to the Committee for cooperative studies. The following procedures were tentatively agreed upon:

1. Gravimetric determination as zinc pyrophosphate.
2. Convert all pyrophosphate to ortho-phosphate by acid treatment and determine total P₂O₅ by the
official A.O.C.S. method. (Since this method will not differentiate between pyro- and ortho-phosphate, it is included only as a check on the method No. 1 above).

3. Moisture content will also be determined so that all results reported may be calculated to a comparable basis.

**CARBONATES AS CO₂** The Committee agreed that further work was necessary on the method proposed by Messrs. Hitchcock and Divine. The chairman appointed a sub-committee to study the proposed method further. The modified procedure will be submitted to this group for cooperative tests.

**Method B. Rosin, (McNicoll Method)**

### Apparatus

- **Esterification Flask.**—A 150-ml flask of either the round-bottom or Erlenmeyer type shall be used.
- **Reflux Condenser.**—Any suitable water-cooled, glass reflux condenser may be used.

### Special Solutions Required

- **Potassium Hydroxide (0.2 N).**—Accurately standardize a 0.2 N solution of KOH in neutral redistilled 95% ethyl alcohol (due to volatility of alcohol, this solution should be standardized frequently).
- **Naphthalene-β-Sulfonic Acid Solution.**—Dissolve 40 g. of Eastman grade or equivalent reagent in 1 liter of c.p. absolute methyl alcohol.
- **Phenolphthalein Indicator.**—Prepare a 0.5 per cent solution in neutral redistilled alcohol.

### Procedure

(a) **Preparation of Fatty and Rosin Acids.**—For the preparation of the sample for this determination, follow the procedure described in C-VII under “Preparation of Total Fatty Matter.”

(b) **Esterification and Titration.**—Weigh about 2 ± 0.001 g. of the fatty acids into the esterification flask. Add 25 ml. of naphthalene-β-sulfonic acid solution. Add a few glass beads to ensure smooth boiling, attach the reflux condenser, and boil for 30 min.; also, run a blank test using 25 ml. of the reagent. At the end of the boiling period cool the contents of the flask, add 0.5 ml. of phenolphthalein indicator, and titrate immediately with 0.2 N alcoholic potassium hydroxide.

(c) **Calculations.**—Calculate the results as follows (Note 1):

\[
R = \frac{(S - B) \times N \times 0.346 \times 100}{W}
\]

\[
R_1 = R - 1.0
\]

\[
R_2 = \frac{R_1 \times F}{100}
\]

\[
R_s = \frac{R_1 \times 1.064 \times A}{100}
\]

where:

- \( R \) = percentage of rosin in fatty acids,
- \( R_1 \) = corrected percentage of rosin in fatty acids (Note 2),
- \( R_2 \) = percentage of rosin on basis of original sample,
- \( R_s \) = percentage of rosin soda soap on basis of original sample,
- \( S \) = milliliters of KOH required to titrate sample,
- \( B \) = milliliters of KOH required to titrate blank,
- \( N \) = normality of KOH,
- \( W \) = weight of sample,
- \( F \) = percentage of total fatty acids in soap, and
- \( A \) = percentage of total anhydrous soap.

If true fatty acid soap is desired, subtract the rosin soap from the total anhydrous soap.

**NOTE 1.**—In all cases where the rosin content is found to be less than 5 per cent, the actual presence or absence of rosin should be checked qualitatively by the Liebermann-Storch test, which may be found described in detail in the note under Wolff’s Method, Modified.

**NOTE 2.**—Cooperative studies have shown that the McNicoll method gives results approximately 1 per cent higher than the amount of rosin present. Consequently, the committee recommends deducting 1 per cent from the percentage of rosin found in the fatty acids.

### PREPARATION OF TOTAL FATTY MATTER (Fatty and Rosin Acids, and Unsaponified Matter)

**Special Solutions Required**

- **Sulfuric Acid (30 per cent).**—Slowly add 650 g. of H₂SO₄ (sp. gr. 1.84) to 1400 ml. of water.

**Preparation for Rosin and Titer Tests, Iodine and Acid Numbers**

Dissolve about 50 g. of the sample in 500 ml. of hot water. (If soaps to be tested contain alcohol, the alcohol should be completely removed by evaporation from the soap solution.) Add 100 ml. of H₂SO₄ (30 per cent), heat gently until the fatty matter collects in a clear layer. Siphon off the aqueous acid layer, add 300 ml. of hot water, boil gently for a few minutes, and siphon off the aqueous acid layer. Wash the acids in this manner three times. Complete this acidification and washing in a very short period of time, and keep the beaker covered to prevent oxidation of the acids. After the last washing, remove the last traces of water from the beaker with a pipette, filter the fatty acids through one or two thicknesses of filter paper, and dry at a temperature of 105°C. for 45 to 60 min. These acids may then be used for the titer and rosin determinations.

In preparing the acids for the iodine and acid number determinations, the washed acids should be filtered through one or two thicknesses of filter paper at a temperature not exceeding 15°C. above the titer point of the fatty acids. If the acids are not perfectly clear and dry, refilter.

### IODINE NUMBER (WIJS METHOD)

**Special Solutions Required**

(a) **Wijs Iodine Solution.**—Dissolve 13.0 g. of re-sublimed iodine in 1 liter of c.p. glacial acetic acid and pass in washed and dried chlorine gas until the original thiosulfate titration of the solution is not quite doubled. There should be no more than a slight excess of iodine, and no excess of chlorine. When the solution is made from iodine and chlorine, this point can be ascertained by not quite doubling the titration (Note). For preparation of the Wijs solution use glacial acetic acid of 99.0 to 99.5 per cent strength. For glacial acids of somewhat lower strength, freezing and centrifuging or draining, as a means of purification is recommended. Preserve the solution in amber, glass-stoppered bottles, sealed with paraffin until ready for use. Mark on the bottles the date on which the solution is prepared; do not use Wijs solution that is more than 30 days old.

**NOTE.**—For preparation of the solution, McIlhiney gives the following details:

*Wolff’s Method, Modified, to be designated Method A.*