<sup>14</sup>Kuhn, R., and Brockmann, H., Z. physiol. Chem., **206**, 47 (1932); Ber., **66**, 407 (1932). <sup>16</sup>Tswett, M., Ber. deut. bot. Ges., **24**, <sup>16</sup>Stout, A. W., and Schuette, H. A., and Fischer, R. A., J. Am. Chem. Soc., 56, 210 (1934). <sup>17</sup>Gill, A. H., J. Ind. Eng. Chem., 10, 612 (1918). <sup>18</sup>Zechmeister, L., and Tuzson, P., Z. physiol. Chem., 225, 189 (1934).

<sup>19</sup>Boekenoogen, H. A., Rec. trav. chim., 56, 351 (1937). <sup>20</sup>Strain, H. H., J. Biol. Chem., **105**, 523 (1934). <sup>a</sup>Winterstein, A., and Stein, G., Z. physiol. Chem., **220**, 247 (1933).

# **1937 REPORT** A. O. C. S. COMMITTEE ON SOAD WRAPPER PAPER

BALLOT was sent to members of this committee late in August to ascertain their opinion concerning methods of testing or evaluating soap wrappers, on which this committee has done collaborative work in previous years.

Replies were received from eight of the twelve members of the committee.

#### Summary of Replies to Ballot:

1. Do the collaborative results to date justify, in your opinion, the recommendation by this committee of any tentative method for evaluating soap wrap paper?

Yes-4; No-3; Not voting-1 2. Are you in favor of doing more collaborative work before submitting any tentative methods to the American Oil Chemists' Society?

Yes-3; No-4; Not Voting-1 3. Which method or methods do you favor to be used in evaluating soap wrap papers?

· · · · · ·	· · · · · <b>F</b>	I T T T		
(a)	Alkali	spot tes	t only.	 0

- (b) Extraction test only...... 1
- (c) Soap contact test only..... 2
- (a+b) Spot & extraction tests.. 0 (a+c) Spot & soap contact tests 1
- (b+c) Extraction & soap contact
- tests ..... 3 (a+b+c) Spot & extraction &
- contact tests ..... 1

4. Additional comments and recommendations:

# W. H. Burkhart:

"Although much work remains to be carried out on this problem the results obtained in my opinion jus-tify the issuance of the methods tested, as tentative standards. Such publication should promote wider discussion and experimentation."

# T. Linsey Crossley:

"After the members of a committee have done work on a committee project, they tend to a sort of short-sightedness to flaws in their processes. Hence having laid out a method and put it through its paces, it seems advisable to turn it loose among the members at large, and the best way of doing that is to propose it as a tentative standard, subject to revision after a year or so."

"My personal feeling is that the spot test should not be recognized."

"With reference to the notes made by two members of the committee as to difficulty in yellow and red elements in the extraction method, I did a lot of work trying to make a scheme that would match each case, but found that it would require a separate combination of tints for every type of paper and sometimes for two lots of the same type. Hence I came to the conclusion that it was not practical and further that intensity was far more readily noted than exact tint match. In the case of the Nesslerizing of water there is often this tint difficulty, but only in the cases where the figure is very low, and a similar thing happens with very small amounts of chlorides and silver chromates."

## Frank Libby:

"There is a very good article relating to this subject under the title of 'The Discoloration of Soap-Wrap Paper' by W. R. Keating. This was published Sept. 2, 1937, in Volume CV No. 10, of the Paper Trade Journal. The paper was originally presented at the February meeting of T. A. P. P. I. in New York."

#### A. S. Richardson:

"I think that A.O.C.S should hesitate to give formal approval to performance tests as distinguished from methods of analysis and of determining physical and chemical constants. The word 'evaluating' as used above compels me to vote "no" on question (1) and refrain from voting on question (2). I do not mean to question the value of the work of our committee, results of which can be published in OIL & SOAP to be used at discretion of the reader. I prefer 'extraction' test (b), which I think that we can sponsor properly as a chemical test without calling it a method of evaluation."

## H. L. Roschen:

"All three methods appear to rate the several papers in much the same order, the soap contact method

giving best results. I am almost inclined to vote for submission of the contact tests as tentative methods; however, I object to the method of grading the results as 'slight,' 'marked,' etc. If we submit a method permitting such designations, it is apt to give rise to difference of opinion on the part of buyer and seller due solely to differences in idea of what may constitute 'slight' or 'marked.' So before we submit methods, I should like to see more coöperative work. Perhaps some standard colors or stains should be introduced, or perhaps the contact methods could be combined with the spot test method, reporting the color produced in the contact test as equivalent to the color of N/10, N/2, N, 2N alkali on the same paper.

"Since it is a quantitative method, I should like to see the extraction test adopted. However, I feel that the agreement between collaborators on the same sample has not been sufficiently good and that the method which we used might be improved. I believe it would be well to work on the T.A.P.P.I. method, using more dilute color solutions so that greater spread in numerical values will be obtained, and also introducing a cold filtration of the This latter step would extract. eliminate difficulty with suspended matter from the paper, and might obviate the difficulties which some members of the Committee have had with milky or cloudy solutions. I am assuming that the cloudiness and milkiness is due to wax on paper K-30 W-35 and that this could be removed by cold filtration of the extract."

# M. L. Sheely:

"We agree with your comments cocerning the extraction method. We believe that further collaborative work is necessary on this method before it should be adopted even tentatively."

#### Conclusions

The votes indicate a small majority in favor of recommending tentative methods for testing soap wrap paper.

Analysis of the replies to question No. 3 of the ballot as to which method were preferred shows that there were only 2 votes for the spot test (to be used in conjunction with other tests) whereas there were 5 votes involving the extraction test and 7 votes involving the soap contact test, alone or in conjunction with other tests.

The spot test does not appear to warrant consideration as a tentative method of the A.O.C.S.

In view of the fact that the Technical Association of the Pulp and Paper Industry has already adopted as a Tentative Standard an extraction method for testing soap wrap paper which differs only in minor details from the extraction method used in collaborative work by the A.O.C.S. in 1936, it would seem advisable that this committee publish as part of its report the T.A. P.P.I. Extraction Method, but without recommending it as a Tentative Method of the A.O.C.S. for another year. Meanwhile members of this committee and others interested can try out this method, of which details are as follows:

The Committee on Standards of the Technical Association of the Pulp and Paper Industry approved as a Tentative Standard the Extraction Method developed by the Committee on Soap Wrap Paper of T.A.P.P.I., and reported at the February, 1937, meeting of T.A. P.P.I.

#### T. A. P. P. I. Extraction Method

The method was published in the Paper Trade Journal of May 13, 1937, page 50.

"Alkali Staining Property of Paper" It is desirable that certain papers, particularly those used as soap wrappers, should not stain unduly with alkali. In the past this property has been generally judged by dropping upon the paper alkaline solutions of varying strengths. The present method is designed for a quantitative statement of the resistance to alkali staining.

## Reagents

1. Potassium Bichromate Solution. Dissolve 0.25 gram of K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> in a small amount of water and dilute to 1 liter.

2. Congo-red Solution. Make up a 1% solution in water. Eastman Kodak Company Congo Red E. K. 770 is recommended.

3. Normal Sodium-Hydroxide Solution.

#### **Apparatus**

1. Nessler Tubes. A set of six is convenient. They should be made with thin colorless walls 1 mm. thick, have a diameter of 29-30 mm., and the 50 cc. mark should be about 90 mm. from the bottom outside.

2. Precision Pipette: Capacity 1 cc., graduated to 0.01 cc.

# **Test Specimen**

The test specimen shall consist of 3 grams of the paper torn into pieces  $\frac{1}{4}$  to  $\frac{1}{2}$  inch square (see Note No. 1).

#### Procedure

Place the test specimen in a 250 cc. Erlenmeyer flask, add 50 ccs. of hot water and boil for 5 minutes. Decant off the liquid into a 100 cc. flask or graduated cylinder. Add 50 cc. of hot water to the paper in the flask and boil again for 5 minutes. Decant the second liquid into the first and make up to 100 cc., using the make-up water to wash the paper in the flask.

Add 25 cc. of normal NaOH solution to the combined liquids and, after standing for at least 5 minutes, filter, using a fast filter paper if the insoluble matter is floccy, or a close filter if it appears cloudy. Compare the clear filtrate with standards made as follows:

To 50 cc. of the bicromate solution add 0.1 cc. of the congo-red solution and mix well. Place dif-

ferent amounts of this mixed solution, such as 0.2, 0.5, 1.0, 2.0 ccs., each in separate Nessler tubes, dilute to the 50 cc. mark with water and mix.

Place 50 cc. of the filtered alkaline solution from the paper in another Nessler tube and compare with the standards. The solutions are best compared by holding the tube containing them over white paper, but not resting on it, and looking down the inside of the tube. If none of the standards match the solution, make up standards of other strengths and compare them with the solution.

#### Report

Report the number of ccs. of bichromate-congo-red solution required to match the tint of the alkaline extract of the paper as the alkali-staining number. Express results to 1 decimal place.

## Interpretation of Results

It has been found that papers showing an alkali-staining number of more than 3 will give a marked stain with 1 per cent caustic soda solution by the drop test.

## Notes

1. With vegetable parchment a finer subdivision of the sample is advisable.

2. Exact matches of tint are not always possible, but distinction in intensity is readily seen.

3. As bichromate slowly destroys the congo-red color the solution should not be mixed until immediately before use.

## **Soap Contact Test**

The affirmative votes involving the use of a soap contact method for testing soap wrapper paper appear to warrant that this method as published in OIL & SOAP in the 1936 report of the Soap Wrapper Committee be recommended as a tentative standard of the A.O.C.S. L. F. HOYT,

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