

not available, a larger sample should be used to obtain more accurate spectrophotometer readings, especially at very low uranine concentrations.

Collaborative results do not show whether the fluorescent nature of uranine introduces errors large enough to account for the difficulties. All results, however, definitely indicate that a reagent blank is essential for accurate analyses.

Although for most spectrophotometric analyses the maximum absorption peak is optimum, the Associate Referee's work indicated that best results for uranine were obtained on the shoulder of the spectral curve.

It is recommended* that further investigative work be done on the proposed method for uranine.

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I. Hanig, H. Kohnstamm and Co., Inc., Brooklyn, N. Y.

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No reports were given on heavy metals, identification, inorganic salts, intermediates derived from phthalic acid, intermediates in triphenylmethane dyes, lakes and pigments, non-volatile unsulfonated amine intermediates, spectrophotometric testing subsidiary dyes in FD&C colors, sulfonated amine intermediates, unsulfonated phenolic intermediates, or volatile amine intermediates.

* For report of Subcommittee B and action of the Association, see *This Journal*, 40, 26 (1957).

REPORT ON MICROCHEMICAL METHODS

By C. L. Ogg (Eastern Regional Research Laboratory,*
Philadelphia 18, Pa.), *Referee*

Microchemical methods for iodine and for phosphorus were investigated during the past year. In these studies the collaborators were provided with tentative methods and test samples for both elements.

Because the determination for iodine is similar to that for chlorine and bromine, 37.1-37.4, the usual preliminary study was omitted. Instead,

* A laboratory of the Eastern Utilization Research and Development Division, Agricultural Research Service, U. S. Department of Agriculture.

the official method for chlorine and bromine was modified to make it applicable to the analysis for iodine, and the modified procedure was tested collaboratively. The results were not as good as those obtained by the present official methods for other elements. In general the agreement between the collaborators' means and the theoretical value was acceptable, but too frequently the intralaboratory precision was poorer than the Referees consider acceptable for an official method. Study of this method should be continued.

A preliminary study of methods for phosphorus was reported at the 1955 meeting. The results from this study were made the basis for the method tested this year. The accuracy and precision of the collaborators' results were satisfactory.

In addition to the collaborative studies, a questionnaire was sent to the collaborating microchemists to get their opinion for future studies on microchemical methods. They were asked to check the microanalytical determinations which they believed should be included in future studies. Thirty completed questionnaires were returned.

Following is the list of determinations and the per cent of the collaborators who checked each determination: molecular weight, 57 per cent; oxygen, 50 per cent; moisture, 43 per cent; hydroxyl group, 40 per cent; neutralization equivalent, 33 per cent; active hydrogen, 30 per cent; hydrogen number, 16 per cent.

These determinations were added to the list by the collaborators: fluorine, 10 per cent; saponification equivalent, 7 per cent; high temperature combustion, 3 per cent; C-methyl groups, 3 per cent; and nonaqueous titrations, 3 per cent.

To determine the number of collaborators who use the newer Shelberg or Zimmermann procedures, the collaborators were asked to designate the type of Dumas procedure they were employing. Twenty-two used the Pregl method, two the Shelberg, and five the Zimmermann. When the last studies of this method were made, the results were about the same. It had been hoped that more analysts were using the newer methods so that a good collaborative test of these methods could be made.

The questionnaire also included a request for suggestions on methods or modifications of methods for the acetyl determination. In the preliminary study of this determination none of the commonly used methods seemed very promising as a potential official method. Three collaborators had suggestions about this determination, and it is hoped that they will help to develop a satisfactory method.

The alkoxyl method adopted as first action in 1955 was based on a well-established method which had been used successfully for years. Only two changes of consequence were made in the method as a result of the three collaborative studies prior to its adoption as first action. Because both changes improved the accuracy and precision of the method, and because

it has been used successfully for a year by a number of the collaborators, the Referee feels justified in recommending that it be adopted as official.

It is recommended*—

- (1) That the method employed in this year's study of the phosphorus determination be adopted as first action.
- (2) That the study of microchemical methods for iodine be continued.
- (3) That studies of methods for the acetyl group be continued.
- (4) That the determination of molecular weight be studied.
- (5) That microchemical methods for the determination of oxygen be studied.
- (6) That studies be continued on the Dumas method for nitrogen.

* For report of Subcommittee C and action of the Association, see *This Journal*, 40, 32, 33 (1957).

REPORT ON MICROANALYTICAL DETERMINATION OF IODINE

By AL STEYERMARK (Hoffmann-La Roche Inc., Nutley, N. J.),
Associate Referee

As a result of previous collaborative studies (1,8,9) on the determination of bromine and chlorine, a method for these halogens was adopted as first action (9). This method has also been used successfully for the determination of iodine in the author's laboratory during the past seventeen years. Iodine was not included in the previous studies, so it was decided this year to have the collaborators analyze an iodo-compound by the halogen method with the slight modification, necessary for iodine, of increasing the time of digestion of the silver halide. The method used is as follows:

CARIUS COMBUSTION METHOD¹ (5, 9)

REAGENTS

- (a) *Fuming nitric acid*.—Reagent grade, halogen-free, sp. gr. 1.50
- (b) *Silver nitrate*.—Reagent grade, powder.

APPARATUS

(a) *Combustion tubes* (5,6).—Use clean, 240 ± 10 mm long by 13 ± 0.7 mm o.d. standard wall Pyrex tubes or 210 ± 10 mm long by 13 ± 0.8 mm o.d. Pyrex tubes with 2.3 ± 0.3 mm walls (see tabulation), free from flaws and with rounded seal at the bottom (*Official Methods of Analysis*, 8th Ed., 37.2 a).

¹ To alter the conditions (temperature, size of sample, volume of acid etc.) might present an explosion hazard.