

Report Of Committee On Indicators

THE work of the Committee on Indicators this season has been divided into two parts, namely:

1. A continuation of the study of an alternate indicator for free fatty acid tests along the lines of our recommendations last year.
2. An investigation of the possibility of substituting Isopropyl Alcohol for Formula-30 in the present free fatty acid test.

It will be remembered that our last report recommended only one indicator, Dr. Grubler's Aniline Blue, for further study as an alternate. This indicator is available in alcohol and water soluble form. Both were carefully studied in Formula-30 and Isopropyl Alcohol with regard to their clearness and sensitivity of end point, CO₂ absorption, and agreement with the present standard method. From this study the conclusion was reached that alcohol soluble Aniline Blue in Isopropyl Alcohol is most desirable and a satisfactory alternate for the present test. Other combinations of these indicators and alcohols all showed undesirable characteristics to the extent that they offered no advantages over the present method.

The combination of alcohol soluble Aniline Blue and Isopropyl Alcohol seemed to satisfy our requirements completely. This solution requires only slight additions of base for neutralization and is affected by atmospheric CO₂ very little, maintaining a neutral color over a considerable period in an open flask. The end point is very sharp, the color changing from the characteristic green or blue-green in the acid solution to a strong red with from .05 to .1 cc of .25 normal base even on dark oils. This is of advantage since the titration may be carried to a distinct red thus eliminating errors due to intermediate shades. The results obtained using this indicator solution check very closely those obtained by the standard method. There was no detectable tendency for results to be either higher or lower than the control. In studying this phase the committee determined the free fatty acid of the majority of the check seed samples by both methods. In no case, on this series or on other data submitted, was

there an instance in which the Aniline Blue result was out of check range as compared with the standard method when a split sample of oil was used.

In a previous study of the suitability of Isopropyl Alcohol for free fatty acid tests (*) a suggestion was made of the possibility of the Isopropyl Alcohol tending to promote saponification, thus giving high results on oils of high acid content. Mr. C. H. Cox studied this point closely using oils of theoretic acid values in ranges up to fifteen percent acid and found no confirmation of the suggestion. The committee also found no variation in the results of high acid oils up to twenty percent acid between the Aniline Blue-Isopropyl method and the standard.

The most desirable concentration of Aniline Blue seemed largely a matter of personal choice. The majority of the committee concluded however that .025% gave the best color change and one in which the approach of the end point was most easily discerned. On exceptionally dark oils there is a possibility that the end point might be somewhat obscure using this concentration. In such cases a .1% concentration will give such a deep color that the oil color is completely masked allowing the end point to be recognized.

It was the original intention of this investigation to discover a suitable alternate to be used on dark oils only, however the close checks obtained and the fact that Isopropyl Alcohol is a much better solvent for fatty oils than Formula-30 and thus requires a great deal less shaking during titration, has led to the conclusion that it is equally satisfactory on any oil. No work was done on refined or other very low acid oils. There was however some correspondence on this point with the Fat Analysis Committee and that phase of the subject was turned over to them for their consideration and report.

This committee also favors retaining the use of 10 cc. of petrolic ether to be added to the oil before the alcohol solution, where it is now stipulated in the methods.

The fact that there are no restrictions on the purchase of Isopropyl Alcohol and that it is a superior solvent for oil caused the

committee to investigate its substitution for Formula-30 in the present test. Results checking with the control were obtained consistently and no objectionable characteristics noted. It was thought that this information would be of particular interest to those laboratories using alcohol almost solely for the free fatty acid test.

It has always been the policy of the Society to eliminate alternate procedures or methods of manipulation on any test in the belief that inter-laboratory checks are more easily obtained when a standardized routine is used. Therefore, it is with some hesitation that this committee makes the following recommendations which will permit three more or less distinct variations of procedure on the free fatty acid test. However, the need for a different indicator on certain oils and the suitability of the proposed methods seems to justify our position, at least until general usage by the Society proves one of them superior. The Committee on Indicators recommends the following tentative changes in the methods governing the free fatty acid test:

1. .025% Alcohol soluble Aniline Blue in Isopropyl Alcohol shall be designated as an alternate indicator solution to be used on the free fatty acid test on all Crude Cottonseed Oils, retaining the use of 10 cc. petrolic ether added to the oil before the alcohol solution where it is now stipulated in the methods.
2. The Secretary of the Society shall be instructed to purchase a supply of Dr. Grubler's Aniline Blue for sale to the membership.
3. Isopropyl Alcohol shall be a permissible substitute for Formula-30 alcohol in the present test.
4. The present Committee on Indicators shall be discharged.
J. L. Mayfield, Chairman
P. McG. Shuey
Dr. F. D. Snell
R. R. Haire
E. C. Ainslie
J. R. Mays, Jr.
A. G. Bedell

(*) Dr. H. A. Schuette: Isopropanol as a substitute for Ethanol. *Industrial & Engineering Chemistry* — Vol. 18, No. 12, December 1926.