

Report of the Committee on the Determination of Soap in Refined Oil 1941-1942

The Committee has not worked on coöperative samples during the past year. It was decided that until some reasonable explanation could be found for the rather poor checks obtained by the coöperative laboratories using the best methods that had come to the attention of the Committee further work on coöperative samples would be futile.

Research on methods for the determination of soap in refined oil has been conducted in at least two of the laboratories represented on the Committee, and out of this work some suggestions for further coöperative work have developed.

It has been found that it is extremely difficult to prepare stable samples of oil containing soap, especially at higher concentrations of soap. The soap tends to flocculate out; and it is extremely difficult, if not impossible, to take a representative sample from the mixture of oil and flocculated soap. This may explain some of the difficulties experienced in the past work of the Committee. To eliminate this difficulty it has been suggested that in future coöperative work samples be prepared by each coöperating laboratory and the freshly made samples analyzed.

The determination of soap in refined oil consists essentially of two steps. The first consists of either an extraction or a concentration of the soap or of the metallic ion. The second consists of the quantitative determination of the metallic ion (almost invariably the sodium ion) or of an anion associated with it.

Obviously no check can be made on the efficiency of extraction unless a suitable method for the determination of the extracted soap or metallic ion is developed. The Committee, therefore, plans first to develop a suitable method for determining the metallic ion. Two and possibly three methods have been proposed which appear to be more sensitive and more easily reproducible than those previously tried by the Committee.

H. A. Boekenoogen (1) has suggested that the sodium ion be determined by the method of Kahane and Dumont (2). This is a gravimetric method involving the weighing of a sodium magnesium uranyl acetate. This compound has a molecular weight of 1532.5, and therefore there is a factor of .198 in converting to sodium oleate. The method has been tried in one of the coöperating laboratories and with certain changes may prove very satisfactory. The universal presence of sodium is a disadvantage, and great care must be exercised or large blanks result. The Committee proposes to study this method. A copy of the method to be studied may be obtained by addressing the Chairman.

R. G. Zehnpfennig has suggested a colorimetric method for the determination of the chloride ion which

is determined in the Durst (3) and Stillman-Durst (4) methods. The method is adapted from one suggested by F. D. Snell (5) (6). The method consists essentially of shaking the chloride solution with silver chromate and determining the chromate ion liberated colorimetrically. It has been demonstrated that this method is capable of a considerably higher degree of accuracy than the method involving the titration of chlorides with silver nitrate using a chromate indicator. The Committee plans to study this method. The details of the method may be obtained by addressing the Chairman.

In the ashing procedure the soap is burned to sodium carbonate and the amount of soap is usually determined by dissolving the ash in water and titrating the hydroxyl ion. One of the members of the Committee, A. Kiess, suggests that methyl orange plus 1 per cent cyanol be used as the indicator.

Should the Committee decide on a suitable method for the determination of the metallic ion or the associated anion, extraction methods will be studied. The Stillman-Durst (4) or a similar method such as that suggested by Boekenoogen (1) have the advantage of simplicity and ease of manipulation. The ashing method, while rather messy and requiring platinum ware, is certainly feasible.

We feel hopeful that a satisfactory method can be worked out in a reasonable period of time. We therefore suggest that the Committee be continued for another year.

The Committee acknowledges with thanks the assistance of R. G. Zehnpfennig, of the Research and Technical Department of Wilson & Company, whose observations on preparation of samples and methods for the determination of sodium and chloride ions have been very helpful.

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