

to the use of a slightly lower temperature. On the other hand, our values are somewhat higher than those proposed by Hilditch and Murti, These investigators noted "low" thiocyanogen values for acids which had been stored for some time, a phenomenon which we have also observed, more especially with some of our earlier specimens of acids. In specimens carefully preserved in evacuated sealed ampules at -20° , we have usually had no difficulty in this respect. It is difficult to see how an acid may change during storage without fall in iodine number and still show a drop in the thiocyanogen value.

We may conclude from our results and from those reported from other laboratories that the thiocyanogen method is a valuable aid in fat and oil analysis but it is clear that the method should be studied further with the objective of setting up standardized details of procedure which can be agreed to by the investigators interested in this technic. Reference samples of acids should be prepared preferably several specimens of each, and results of analysis from different laboratories compared. The official Committees on fats and oil analysis of the American Chemical Society and the American Oil Chemists Society might well undertake such a cooperative problem.

Summary

The thiocyanogen reagent may be stabilized by storage at temperatures of 3° or less. The reaction of thiocyanogen with highly purified linoleic and linolenic acids has been studied under many variations of experi-

mental conditions. Reaction rates of thiocyanogen with oleic, linoleic and linolenic acids at 3° , 16° and 25° and with variable excess of reagent are described. Oleic acid gives a maximum thiocyanogen absorption which is apparently equal to the iodine number, within 3-6 hours. The reaction with linoleic acid is rapid up to 3 hours and shows a slow constant increase thereafter. A modified procedure of analysis, similar to the official method, employs a 0.2 N thiocyanogen reagent, containing 10 percent carbon tetrachloride and a reaction temperature of 16° . Results of analysis of several specimens of acids by several preparations of reagent shows an average thiocyanogen value of 96.6 for linoleic acid and 166.3 for linolenic. Simultaneous equations, derived from these values, were used in the analysis of five mixtures of known composition with excellent results.

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Report of the Olive Oil Committee 1940-41

THIS committee has had under consideration two matters: first, the application of the approved Fitelson Test for the detection of teaseed oil in olive oil to those olive oils giving interfering colors and secondly, the value of the U.S.P. cold test on edible olive oils.

We found, in our work on the Fitelson Test, that the color of some olive oils, especially the inedible or so-called commercial olive oils, as a class, tended to mask the characteristic reddish coloration developed by ether in the final stage of this test. To overcome this difficulty, the following modification, suggested by the chairman, has been tried, found satisfactory, and is recommended for adoption as an alternative when conditions are met with which show the necessity for clearer colors.

"Saponify 5 grams of the oil to be tested plus 5 grams of colorless mineral oil or "albolene" with excess alcoholic potash (or 5 cc. of 50% KOH in about 30 cc. of alcohol). Brisk boiling for from 10 to 15 minutes usually suffices. Pour the liquid into a separatory funnel, add an equal volume of water, shake and allow the layers to separate. Draw off and reject the lower soap solution. Wash the oily layer with water several times to get rid of the soap. Finally dry the oil over anhydrous sodium sulphate and filter for the test. Seven drops of this oil are used in the regular way for the Fitelson test, using, however, standards for comparison made up with mineral oil in the same way, which can be kept permanently."

The second matter under investigation was a result of numerous requests from members of the olive oil trade because several rejections of olive oil occurred in the past few years by reason of the cold test in spite of the fact that all other requirements were met.

The U. S. P. XI on page 263, under tests for identity

and purity (!) of the olive oil, states that "when cooled to from 10° to 8° C., olive oil becomes *somewhat* cloudy and at about 0° C. it *usually* becomes a whitish granular mass." (The italics are mine.)

This test has been in the Pharmacopeia since 1880 without change. Whether conditions of manufacture have actually altered the situation which may once have existed, or, perhaps because the test in spite of its loose wording is being applied seriously, the fact remains that some undoubtedly pure olive oils have been turned down on this test alone.

The test give no time limit so that a reasonable length of time must be read into it. It is also placed in the category of a purity or identity test, which is rather difficult to understand as "cold" tests are usually considered indices of quality or grade. This is the case with winterpressed cottonseed oil, for example.

However, the committee took the matter up seriously and some of the members have tried the cold test on quite a number of oils. The consensus of opinion is that the test is worthless as a test for identity and purity of olive oil and that from any standpoint it is of no particular value since the olive oils themselves vary so greatly in composition.

OLIVE OIL COMMITTEE:

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