Simplification of The Wheeler-Swift Stability Test*

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THE testing method for evaluating fats and oils for keeping quality discussed in this paper has already been adequately described (1 to 4). In the modification of Stebnitz & Sommer[4] less dependence is placed on the sense of smell in determining the end or rancid point of the aeration, inasmuch as this is indicated by color changes occurring in tubes containing very dilute alkali and methyl red, into which the air bearing the oxidation products from the fat is led. During the course of many experiments made by the present writer involving the effect of light on the duration of the tests, in which the tubes were examined visually, it was noticed in the case of cottonseed products that rather sharp color changes in the fat occurred at the end-point. As the observations continued these changes were used as an aid in determining the end-point. Finally, when reference was made to the work of Wheeler[1], who had followed these color changes quantitatively throughout the course of the aeration of cottonseed and corn oils, it seemed that they might well serve as the principal index of the end-point. The peroxide number determination is simple and easy enough, indeed; but it is felt that where we can eliminate procedures involving weighing on analytical balances, we are contributing substantially to the efficiency of laboratory operation. Especially is this true when large numbers of tests are necessary—and let it be remembered that three peroxide determinations are required for each stability test.

In Figure I is reproduced a pair of curves from Wheeler's paper, one of which shows the sharp break in the color of the oil when it becomes rancid during the aeration. To express the data in terms more familiar to us, Wheeler's peroxide values have been calculated to mille-equivalents per kilogram, and a line has been drawn through the value of 125, which has been adopted by convention as the minimum value at which cottonseed oil may still not be rancid. Just before the rancid point is reached, the oil becomes appreciably darker; then it begins to bleach, during the course of which change rancidity becomes very apparent to the smell, and if the test is allowed to continue, the oil bleaches to a color much lighter than its original. All of the other curves in Wheeler's paper show the same characteristics, and the peak of the color curve in the case of corn oil is very extreme, the color going from 9 to 100, then dropping quickly to a value of 2.

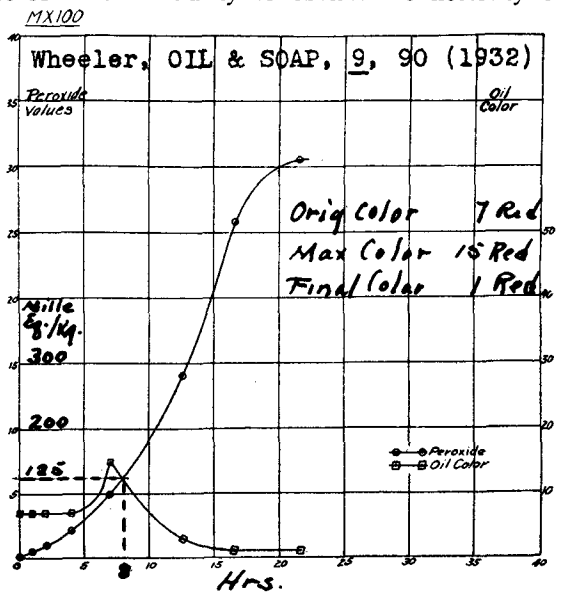

Fig. I—Variation of Oil Color and Peroxide No. with Time of Aeration (Wheeler's Curves).

Apparatus

In order to see if this effect might not be used as a fairly accurate means of determining the end-point without making peroxide number determinations, an apparatus was designed in which the principal feature is a glass bath vessel, permitting visual examination of the tests without the necessity of removing them. The apparatus is shown in Figure II, which, it is believed, illustrates its design with sufficient clarity to obviate the necessity of giving a detailed description. The principal difficulty was to make the vessel vapor-tight to avoid too much loss of water when the tests had to run overnight. This result was attained by cutting a strip of ¼-in. sponge rubber 2-in. wide, and sewing the ends together to form a sort of bushing between the walls of the beaker and the metal collar soldered to the cover. The dimensions were so chosen that the whole cover assembly acted as a reasonably tight-fitting plug or stopper for the beaker. Very little leakage occurred around the oil color tubes in which the tests were made, as they happen to fit fairly closely into standard 1-in. tubing from which the small collars were cut. The internal illumination shown is a convenience rather than a necessity. It was attained by soldering small wires on the terminals of a flash light bulb, which was attached to the end of a piece of glass tubing by means of a short piece of rubber tubing, and the whole supported as shown. The 4 liter Pyrex beaker accommodates 9 tests arranged in a circle.

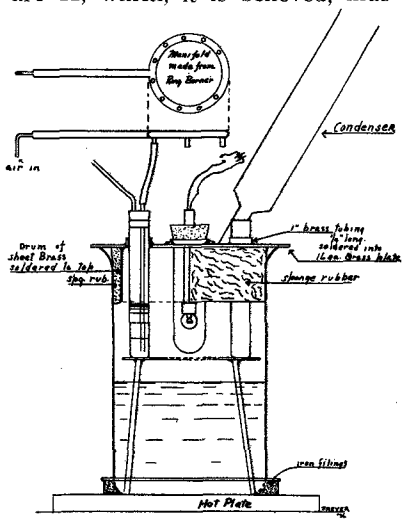

Fig. II—Glass Bath Vessel for Modified Wheeler-Swift Stability Test, Permitting Visual Determination of End Point.

Additional simplification of the

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apparatus was attained by replacing the numerous wash bottles, traps, and manifold bottles by a dry air purifying train. Moisture is removed by calcium chloride; carbon dioxide, by soda-lime; dust, by filtering through cotton; and any hydrocarbon impurities from the air-compression system are removed by granular activated carbon. All these agents are packed in series in two glass towers in the order, calcium chloride, soda-lime, carbon, and cotton.

Effect of Light

Although previous work done in this laboratory has shown that exposure of the tests during aeration to light of ordinary indoor intensity has no effect on the duration of the tests, three runs were made in the glass apparatus to confirm the earlier results. In one run the apparatus was exposed; in the other the beaker was wrapped with an opaque cloth to exclude light. The results were the same in both cases, as shown in Table II.

COMPARISON OF STABILITY TEST RESULTS AS INDICATED BY TWO METHODS OF DETERMINING THE END-POINT OF AERATION.

TABLE II.

Sample	Stability: By Peroxide No. Det'n	Stability: By Visual Inspection of Color Change	Dif.
Oil 1	4.2 hrs.	4.0 hrs.	0.2 hrs.
2	4.7	4.2	0.5
3	6.5	6.0	0.5
4	10.0	9.0	1.0
5	15.9	15.5	0.4
6	15.1	14.5	0.6
7	14.4	15.2	—0.8
8	14.3	15.2	—0.9
9	14.2	15.2	—1.0
10	14.6	15.2	—0.6
11	14.5	15.2	—0.7
12	14.2	15.2	—1.0
13	15.4	16.0	—0.6
14	13.0	13.5	—0.5
15	12.6	13.0	—0.4
16	10.3	10.0	0.3
Hyd'g'd Fat.	69.5	70.0	—0.5
Hyd'g'd Fat.	65.0	66.0	—1.0
Oil A, Light*	10.3	10.0	0.3
Oil A, Dark..	10.4	10.0	0.4
Oil B, Light.	15.3	15.6	—0.3
Oil B, Dark..	15.4	15.6	—0.2
Oil C, Light.	15.5	15.5	0.0
Oil C, Dark..	15.4	15.6	—0.2
		Avg. +,	0.47
		Avg. —,	0.62

(Most of the oils represented in this table contained either catalysts or antioxidants.)

*The following tests show the absence of any appreciable effect of moderate indoor illumination on the accelerated keeping time.

Estimating the End-Point by Visual Inspection

To facilitate the color estimation, 4 samples of oil were adjusted to the Lovibond red color values: 2, 5, 10, and 20, and for simplicity were given the arbitrary numbers 2, 4, 6, and 8. These were found to be convenient reference standards of comparison in the early stages of our trial of this modification and at intervals during the aeration color estimations were made without having to remove any of the tests from the apparatus for colorimeter readings. In Table Ia is given a typical run, but in it the color values are only estimated, relative ones made before the comparison tubes were prepared. The data confirm the results of Wheeler in revealing a darkening of the color just before the rancid point, the on-set of rancidity early in the bleaching stage, and very decided bleaching during the progress of rancidity development. At various stages of the aeration of the three tubes of fat comprising one test, the relative color combinations may be quite varied, and to aid in the interpretation of any one combination when inspecting tests (for example in the early morning after tests have been running overnight), the scheme shown in Table Ib may be considered to represent the ideal case where the tests are permitted to aerate until all of the tubes have become rancid. Ordinarily, of course, the tests are removed after stage 5 is reached; that is, when the tube started first becomes rancid.

PROGRESS OF COLOR CHANGE IN FATS UNDERGOING AERATION IN STABILITY TESTS.

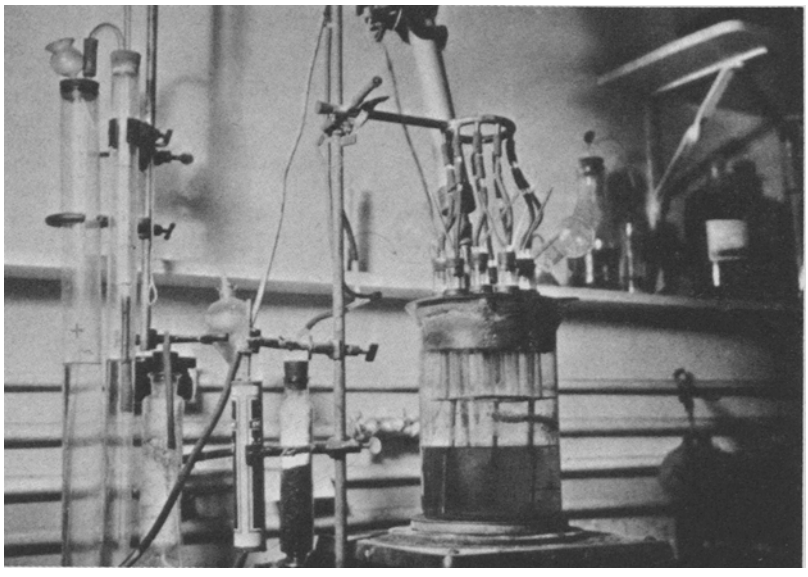
TABLE Ia (Actual)

Time	Test I	II	III	Remarks
	Relative Colors			
Started 6:00 A. M.				Cottonseed compound containing proöxidant
12:00 Noon	4	4	4	
2:00 P. M.	6	5	4	
2:30 P. M.	9	7	5	
3:00 P. M.	10	8	6	
3:30 P. M.	10	8	8	
4:00 P. M.	10	10	8	
4:30 P. M.	8	10	10	
5:45 P. M.	6	8	10	I & II very rancid; terminated test
Hrs. run	11¾	10¾	9¾	
Peroxide No.	174	156	102	Mille Equiv./Kgm.

TABLE Ib (Ideal)

Stage	Time Started: 1	2	3 P. M.	Interpretation
	Relative Colors			
1	4	4	4	All fresh
2	4	4	4	All fresh
3	6	4	4	All fresh
4	8	6	4	No. I starting "out"
5	6	8	6	No. I rancid; should terminate test
6	4	6	8	Nos. I & II rancid
7	2	4	6	All rancid
8	2	2	4	All rancid
9	2	2	2	All rancid
10	2	2	2	All rancid

It should be mentioned that the writer does not consider that the indications of the color changes discussed here are any more accurate or reliable than those of the odors brought from the aerated fat by the effluent air. Some individuals, however, have distinctly impaired olfactory perception, and in any case the color change serves as a useful adjunct, and may be



Showing Use of 4-Liter Beaker as Glass Bath Vessel in Modified Wheeler-Swift Stability Test. Simplified Dry Air Train Is Also Shown.

the sole criterion of the end-point, as indicated below.

During the course of some stability experiments in which it was desired that the results be as accurate as possible, the end-points were estimated visually before making the peroxide number determinations, and in Table II are given the results obtained by the two methods. A respect in which our practice in the use of this test differs from that formulated by King, Roschen & Irwin[2] is that the end-point is taken as the time at which the peroxide value curve crosses the ordinate line for 125 in the case of cottonseed oil, and 75 in the case of hydrogenated fat. This enables closer comparisons to be made in tests differing only by fractions of an hour. Had the keeping times in Table II been determined in accordance with the practice of reporting the nearest whole hour value greater than the threshold value for a given fat, the agreement between the two sets of data would probably have been closer.

For the most accurate work, for example, in experimental studies of antioxidants and proöxidant conditions and materials, it is not suggested that this method of estimating the end-point be used as a substitute for peroxide number determinations; but for refinery control tests, especially when these are quite numerous, this new modification offers distinct time and labor-saving advantages.

REFERENCES

[1]Wheeler, Oil & Soap, 9, 89 (1932).
[2]King, Roschen & Irwin, Oil & Soap, 10, 105 (1933).
[3]Freyer, Oil & Soap, 12, 139 (1935).
[4]Stebnitz & Sommer, Oil & Soap, 12, 201 (1935).

PROPOSED RESEARCH PROGRAM OF THE REGIONAL SOYBEAN INDUSTRIAL PRODUCTS LABORATORY

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THE Bankhead-Jones Act of June 29, 1935, states as one of its purposes—"The Secretary of Agriculture is authorized and directed to conduct research . . . relating to the improvement of the quality of, and the development of new and improved methods of production of, distribution of, and new and extended uses and markets for, agricultural commodities and by-products and manufactures thereof . . . "

In accordance with these and other objectives, the U. S. Department of Agriculture has set up a limited number of specialized laboratories in the major agricultural regions of the country to study some of the broad agricultural problems peculiar to those areas. At a conference of representatives of the Department of Agriculture and the Experiment Station Directors of North Dakota, South Dakota, Nebraska, Kansas, Minnesota, Iowa, Missouri, Wisconsin, Illinois, Indiana, Ohio and Michigan, it was decided to establish a laboratory at the University of Illinois, Urbana, Illinois, for a study of the industrial utilization of soybeans and soybean products. The reasons for reaching such a decision are not hard to find. While the soybean has been an important crop in North China and Japan for thousands of years, and while it has been known in this country for more than 100 years, it is only within the past decade that this legume has assumed an important position in the agricultural and manufacturing industries of the United States. Thus, in 1934 approximately 2,000,000 acres were planted to soybeans, while in 1935 this figure had more than doubled, falling just short of 5,000,000 acres.

In 1925, 5,000,000 bushels of beans were harvested, in 1934 around 20,000,000 bushels, and in 1935 more than 40,000,000 bushels. In 1935, the three states leading in the production of soybeans were Illinois (22,000,000 bu.), Iowa (7,000,000 bu.) and Indiana (6,000,000 bu.). There are at present around 35 mills crushing soybeans, 15 plants engaged in the manufacture of soybean flour, 20 in the manufacture of soybean food products, and more than 50 in the manufacture of other industrial products. While only 2,646,000 pounds of soybean oil were produced in the United States in 1926, it is estimated conservatively that the crush from the 1935 crop might well exceed 200,000,000 lbs., which is more than 5 times the production from the 1934 crop. The production of soybean meal naturally has increased to the same degree. It can be realized readily that the large increase in soybean acreage and the enormous jump in oil and meal production have created actual and potential problems of considerable import to the industry. It would, therefore, seem to be the part of wisdom to develop a well-integrated research program which might solve or anticipate these problems and assist in placing the soybean industry, in all its phases, upon a sound and stable basis.

Naturally the members of the American Oil Chemists' Society are more interested in soybean oil than in other soybean products. Most of you are familiar with its physical and chemical properties. It is classed as a semi-drying oil with an iodine number ranging from 125-137, and a saponification number of 189. Its fatty acids consist of approximately 14% saturated and 86% unsaturated acids. Palmitic acid (7%) and stearic acid (6%), together with small quantities of myristic and arachidic acids, make up the saturated acids. Oleic acid (26%), linoleic acid (55%) and linolenic acid (5%) constitute the components of the unsaturated acid fraction.