

and to Mr. H. S. Bailey, of the Division of Foods, for the preparation of the crystals shown in the cuts.

WASHINGTON, D. C.

THE EFFECT OF HEAT UPON THE PHYSICAL AND CHEMICAL CONSTANTS OF COTTONSEED OIL.

BY ELTON FULMER AND THEO. C. MANCHESTER.

Received July 7, 1908.

For many years, considerable reliance has been placed upon certain color reactions given by fats and oils with various reagents as a means of determining either purity or identity. Some of these tests have been exceedingly useful when applied to normal oils or fats. It has, however, been shown by many workers that the substance to which the color reaction is due is either destroyed or rendered inactive by heat. For example, Halphen's reagent fails to give a coloration with cottonseed oil which has been heated to 250–270°; and if Bechi's test be applied to an oil that has been heated to about 245°, no reduction occurs. The literature covering these points was recently summarized by one of us.

It seemed desirable to know if the application of high temperatures to cottonseed oil would produce changes in the general physical and chemical constants as well as in the color-producing capability, and therefore the work outlined below was undertaken by us. As far as we were able to ascertain there is no reference in chemical literature to this subject except the work of Tortelli and Ruggeri, which is quoted in *Bulletin No. 77 of the Bureau of Chemistry, U. S. Department of Agriculture*. Their conclusions are confirmed by our results as given below.

The following results were obtained:

	Sp. Gr. at 15.5°.				Refractive index at 25°.		Iodine value.	Saponification equivalent.	Free fatty acids calculated as oleic.	
Normal unheated oil . . .	0.9221				1.47509		110.1	191.8	0.06	
Temperature.	Heated to min.	Heated 30 min.	Heated 10 min.	Heated 30 min.	Heated 10 min.	Heated 30 min.	Heated 10 min.	Heated 30 min.	Heated 10 min.	Heated 30 min.
180°	0.9227	0.9228	1.47510	1.47510	110.0	108.1	190.9	190.8	0.053%	0.054%
220°	0.9229	0.9229	1.47518	1.47518	108.8	108.5	190.7	190.2	0.059	0.068
240°	0.9229	0.9236	1.47528	1.47548	108.4	108.5	190.4	190.6	0.130	0.260
250°	0.9236	0.9240	1.47535	1.47563	108.3	107.8	190.6	190.4	0.160	0.400
270°	0.9234	0.9242	1.47549	1.47583	106.9	106.3	190.7	190.9	0.530	0.880

The oil used by us was "winter yellow," the following constants being determined: specific gravity, refractive index, saponification equivalent, iodine value, and free fatty acids. These were determined by the usual methods, Pulfrich's refractometer being used in determining refractive index, and the Hanus method being used for iodine value. The oil was

¹ THIS JOURNAL, 24, 1148 (1902).

heated in test tubes about one and one-half inches in diameter and six inches in length. About 60 cc. of oil were used in each case, the tubes being immersed in a bath of concentrated sulphuric acid and potassium bisulphate.

From these results it appears that, in general, the specific gravity, refractive index and free fatty acids increase, and the iodine value decreases as the temperature of heating increases. The decrease in the iodine value is quite rapid above 180° and is influenced both by the temperature and duration of heating. The saponification equivalent remained practically constant throughout. The refractive index was dependent upon the temperature alone below 220° . Above this point the time of heating affected it more than the temperature. Below 220° there was little change in the free fatty acids. Between 220° and 240° the acid percentage was more than doubled when the oil was heated for ten minutes, and after heating for thirty minutes the acidity was four times as great as in the original oil. At 270° the acidity was nine and fifteen times as great as in the original oil, when heated for ten and thirty minutes respectively. The time of heating above 220° therefore influences the acidity as much or more than the temperature.

While therefore the heating of cottonseed oil does change in some degree its physical and chemical constants, the changes are not sufficiently great to furnish a means of determining whether or not heat has been applied to it, because of the wide limits of these constants in normal unheated oils. In other words, a cottonseed oil which has been heated until its sensitiveness to Halphen's test has been destroyed, and until it no longer reduces silver nitrate, will still give physical and chemical constants well within the limits shown by normal unheated oils, except in case of free fatty acids. However, if heated cottonseed oil should be mixed with olive oil, a determination of acidity would be of no value for proving its presence in the mixture, because the acidity of the latter usually exceeds the percentage found in the former even after heating at 270° for thirty minutes.

CHEMICAL LABORATORY, WASHINGTON STATE COLLEGE,
PULLMAN, WASH.

A METHOD FOR DETECTING SYNTHETIC COLOR IN BUTTER.

By R. W. CORNELISON.

Received July 14, 1908.

In an effort to determine with certainty whether or not a particular sample of butter contained a synthetic dye the results obtained by methods given in the well-known works treating on the subject did not seem to the writer to be sufficiently conclusive. By Martin's method (shaking