

tries. There was a remarkable recovery, however, in 1931.

Denmark is the largest exporter of butter in the world, and it is little realized that a considerable proportion of the production of Danish butter is produced by the feeding of American cottonseed cake and meal, about 80 per cent of our exports going to that country.

#### **Cottonseed Hulls as a Cattle Feed**

The hulls of cottonseed, which for a great many years were a waste product having no use except for fuel, have also become recognized as a valuable cattle feed and are worth consider-

ably more per ton than could be obtained for the seed 25 or 30 years ago. After the seed has been reginned it is run through hulling machines and the hulls removed. Very nearly one-half of the entire weight of the seed is thereby converted into the product known as cottonseed hulls. These—especially when mixed with cottonseed meal—make a cattle feed of superior value, for which there is a steady demand at around \$10 per ton—practically the entire supply being consumed in the Southern states. For the year ended July 31, 1931, 1,303,504 tons were produced, valued at \$10,474,000.

## **Committee Report On Olive Oil**

**M. F. LAURO, Chairman**

*(Reported to New Orleans Meeting)*

**I**N 1927-28, the modified Valenta or Crismer test was primarily responsible for a number of prosecutions in New York City in cases of suspected olive oil adulteration. Interest was thus focused on a test which up to this time had been rarely, if ever, included in the routine analysis of olive oil for purity. A dozen or so court cases, in which the chairman participated, devolved on this and iodine absorption as leading issues of fact, and thus considerable discussion as to the reliability of Crismer values came about.

At about the same time, Dr. Jamieson undertook a study of this method, the results of which are summarized in his recent book on "Vegetable Fats & Oils," 1932.

The test is a simple one and not without its value. Since it had been strongly sponsored by the Olive Oil Association, its official chemists and the local Department of Health, I felt it would be to our interest to have the committee study and perhaps settle the question of its value one way or another.

Accordingly, through relations in Italy, I was able to secure five samples of olive oil of the first pressing from different local presses in the province of Foggia. This oil runs somewhat high in acidity and of pronounced odor

and flavor which mellows out on standing and is usually blended for the general market, but in Foggia finds direct consumption at home.

These samples were sent to the members of the special committee investigating the ultra-violet ray examination of olive oils, to conduct that as well as the Crismer, with instructions to follow the improved procedure outlined in the practical part of Fryer and Weston, "Oils, Fats & Waxes," and in Elsdon, "Edible Oils & Fats."

The ultra-violet ray report was as follows:

Sample No.	Chemist 8	Chemist 2	Chemist 6
1	Virgin	Virgin	Virgin
2	"	"	Virgin
3	"	"	Orange with purple cast
4	"	"	Orange with purple cast
5	"	"	Orange with purple cast

The report of Chemist No. 6 would imply doubt as to samples 3, 4 and 5. Since the oils are undoubtedly pure, this would leave the method open to question; which is about the conclusion reached by other experimenters. See Jamieson "Vegetable Fats & Oils" on the detection of refined olive and other oils.

In the matter of the Crismer test, our second task, while some of the members have not been

---

*In scanning the values for the possible adulterants of olive oil, rape and coconut oils appear to be the only ones with figures sufficiently far removed from those of olive to indicate detection in mixtures.*

---

heard from, there has been sufficient done to warrant a report and I believe, a conclusion.

In brief, this test determines the temperature at which a definite turbidity occurs on cooling a solution of a standardized reagent with the oil to be tested. Valenta, to whom it owes its origin, used acetic acid as the solvent. Subsequent modification and improvement in technique, by Crismer and later by Fryer and Weston have resulted in the present improved form. The reagent is now a mixture of equal parts of ethyl and amyl alcohols, so adjusted by the addition of small portions of water as to show a turbidity at 70° C. with almond oil, when corrected for its acidity. Each oil to be tested has a factor for the class to which it belongs. This factor multiplied by the acidity of the particular oil, must be added to the observed temperature reading to give the true Crismer value.

Considering the small amount of oil (2 cc.) used, it is surprising that results are as definite as they are with the different kinds of oil. Duplicate tests are made on another sample of the same oil, with fresh reagents, and should check within a half degree. Care must be taken to have a dry sample, properly standardized reagent and thermometer, and the procedure must be followed as prescribed. Results are then concordant. These points were brought up in court, as well as the fact that there are as many as eight kinds of amyl alcohol, with impurities in solvent and oil to affect solubility, etc., and explained the discrepancies between values obtained by different operators.

In scanning the values for the various possible adulterants of olive oil, rape and cocoanut oils appear to be the only ones with figures sufficiently far removed from those of olive to indicate the possibility of detection in mixtures with olive oil.

From Fryer and Weston:

<i>Oil</i>	<i>Acidity</i>	<i>Crismer Value</i>
Rape .....	0.60%	83.3
Olive .....	0.7	69.2
Olive .....	1.8	69.2
Olive .....	3.6	69.0
Cocoanut .....	0.0	34.0
Cocoanut .....	1.6	32.2
Almond .....	0.9	70.1

Other likely adulterants of olive oil have values too close, as shown below:

	<i>Fryer &amp; Weston</i>	<i>Jamieson</i>
Sunflower .....	64.0	....
Corn .....	68.2	63.6
Cotton .....	65.2	67.2
Sesame .....	68.1	....
Peanut .....	74.3	62.60, 67.36, 67.78
Lard .....	72.7	....

The committee were to obtain values on the five samples of pure olive oil, and to make mixtures (with them or other olive oils) of rapeseed and cocoanut oils in definite amounts, determine their values and see if the method had any value as a means of detecting the presence of these adulterants.

I did not send any samples to Dr. Jamieson as he had made a study of the method some years ago. So I take the liberty of using his findings on oils of his own, as follows:

	<i>Acidity</i>	<i>Crismer Value</i>
Rape .....	0.14%	82.90
Rape .....	0.51	81.25
Rape .....	0.21	83.88
Rape .....	3.50	82.64
Olive .....	0.35	70.71
Olive .....	0.63	70.88
Olive .....	0.98	70.35
Almond .....	0.65	70.16

His comment is: "As in the case of other characteristics of the oils, such as the iodine number, saponification value, etc., the Crismer test will show more or less variation with a given kind of pure oil from different sources and a range of two or often more degrees is to be expected."

This is instructive, since at the time of the legal actions previously mentioned, too much stress was laid on close agreement with the text values for purity and slight variations were treated as proof of impurity or adulteration. This the writer had to combat with complete analyses on the oils at issue and showed that the Crismer value ranged from 69.8 to 72.5 and even 73 without these oils having any abnormality in other characteristics, or any erucic acid (evidence of rape oil) by the Tortelli and Fortini test. The text value of 69.2 should not be treated as an arbitrary value.

The report of the special sub-committee is as follows:

Oil	Chemist 2		Chemist 3		Chemist 5	
	F. F. A.	Crismer	F. F. A.	Crismer	F. F. A.	Crismer
Olive No. 1.....	3.3%	70.3	3.40%	69.25	3.23%	71.4
Olive No. 2.....	3.2	69.8	3.30	69.1	3.23	71.1
Olive No. 3.....	3.6	69.8	3.57	69.0	3.23	71.1
Olive No. 4.....	2.4	70.3	2.38	69.55	3.23	71.1
Olive No. 5.....	3.6	70.0	3.63	69.0	3.23	70.9
Almond (standard)..	0.28	70.0	2.80	70.0	....	68.1
Almond (standard)..	....	....	....	....	....	70.0
Rapeseed .....	0.20	82.0	0.20	83.3	1.80	77.3
10% Rape/90% Olive	....	71.4 (71.0)*	....	70.4 (70.4)*	....	72.4 (72.0)*
20% Rape/80% Olive	....	72.5(72.24)*	....	72.0 (71.86)*	....	73.0 (72.6)*
30% Rape/70% Olive	....	73.5(73.46)*	....	73.48 (73.3)*	....	....
40% Rape/60% Olive	....	....	....	74.7 (74.72)*	....	....
Cocoanut .....	0.10	32.5	6.05	31.0	10.6	32.2
10% Cnut/90% Olive	....	65.6(66.07)*	....	65.1 (65.2)*	....	67.0(67.5)*
20% Cnut/80% Olive	....	63.0(62.34)*	....	....	....	61.5(63.56)*
10% Cnut/80% Olive/ 10% Rape.....	....	....	....	66.7	....	....

(\*Calculated values.)

#### Miscellaneous samples of Olive oils:

1.0	69.8	4.87%	69.0
1.6	70.3	4.47	69.0
.9	71.4	5.42	69.0
.7	71.9	4.64	69.0
1.2	71.5	1.34	69.0
...	71.8	....	....

Values calculated for the mixed oils from their individual Crismer numbers appear to agree quite closely to those actually obtained.

Including the data from Dr. Jamieson's work, it is seen that rapeseed oil has Crismer values from 77.3 to 83.3, olive from 69.0 to 71.8, and cocoanut from 31.0 to 34.0 (text value). My own examination in 1927-8 on disputed samples of olive oil would indicate possible values of olive oil up to 72.8. These litigated samples had iodine values rather high, 88 to 89, but extensive analysis failed to reveal any adulterant; and in particular, rape oil was looked for and not found present.

Leaving these out of account, it would seem that pure olive oil may range from 69.0 to 71.0.

It also appears from our work, that the Crismer values on other oils vary somewhat from the text (Fryer & Weston):

That a 10 per cent addition of rapeseed oil cannot be said to affect the turbidity temperature sufficiently to indicate its presence;

That a 20 per cent addition might in some cases direct suspicion to the purity of the olive oil, but that at best it would merely indicate and not prove the oil to be impure;

That a 10 per cent addition of cocoanut oil makes an appreciable lowering of the normal values for olive oil, but since the test is not a specific one for this or any other oil, a lower (or higher) Crismer value would simply indicate the olive oil to be impure.

The test is a simple one, easily and quickly made. The reagent once prepared and the apparatus set up, very slight labor is involved. A few minutes' time is all that is necessary to run any number of oils at the one time. For these reasons, the test could very well be included in the routine analysis for olive oil purity. It is of some value as an indication, to be subsequently followed by confirmative tests for the adulterant indicated, such as by the Tortelli and Fortini tests for rape oil, or Reichert-Meissl for cocoanut oil.