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[Received September 19, 1957]

Error in the Sampling of Soap and Detergent Bars for Moisture Determination

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NSTRUCTIONS of the American Society for Testing Materials for the sampling of soap and combination bars specify passing the entire bar through a suitable chopper or, when this is impossible because of the nature of the sample, that a cake be quartered by cutting at right angles in the center and sufficient sample obtained for analysis by shaving equally from all freshly cut surfaces (1).

Sampling by the latter alternative is widely practiced in preference to the chopper method, which is messy and time-consuming. It is based on the assumption that the moisture content of longitudinal slices from the center of a bar approximate the average moisture content of the bar as a whole and that dehydration of the corners of the bar does not affect significantly the average moisture content.

The A.O.C.S. directions for the sampling of bar soap are more precise. They specify grinding of the entire bar or, in the case of large samples, permit quartering of the bars and grinding quarters from each bar. It is further specified that each ground sample should consist of at least three pounds before subsequent reduction to a laboratory-size sample by successive quartering (2).

Griffin $(\hat{3})$ gives a further variation for sampling, recommending the grinding of whole bars or, alternately, cutting each cake diagonally and shaving from the freshly cut surfaces.

A study of the moisture level in all parts of a single bar indicates that an appreciable error may arise in sampling for moisture content unless the whole bar or a symmetric portion thereof, such as a half or full quarter of the bar, is taken for analysis.

The magnitude of the error may be evaluated by determining the moisture content of center slices versus the remainder of the full quarter of the bar. Table I gives results for a series of such comparative determinations.

A simple calculation based on the respective weights of sample taken for analysis of milled soap No. 2 (Table I, first week) indicates that if the whole bar were taken for analysis, the moisture content would be 9.49%, whereas the moisture value obtained by the taking of slices would indicate the moisture to be 10.01%. This difference is considerably greater than the experimental error of the determination, which in the case of bar soap is approximately $\pm 0.1\%$.

The results for soap No. 2 in Table I also show that, as the age of the soap increases, the difference

TABLE I								
Moisture	Content	of	Bars:	Center	Slices	vs.	Whole	Quarter

Aging	Moisture content (oven method)			
before analysis	Shaved slices	Remaining quarter		
1st week 1st week 2nd week 3rd week	$7.53 \\ 10.01 \\ 8.84 \\ 8.75$	$\begin{array}{r} 6.96\\ 9.24\\ 8.55\\ 8.26\end{array}$		
	period before analysis 1st week 1st week 2nd week 3rd week	Aging (oven period before analysis slices 1st week 7.53 1st week 10.01 2nd week 8.84		

in moisture content resulting from the two methods of sampling tends to diminish.

In view of the fact that the result for the determination of moisture in a bar product varies with the method of sampling, it was considered of sufficient interest to determine the moisture content of a bar simultaneously in as many areas as practical. Accordingly 24 samples were taken from one bar of milled toilet soap, and the moisture was determined on a semi-micro scale by the oven method at 105°C. The samples were taken by means of a thin cork borer, and each plug (surface to surface of the bar) weighed about 0.22 g. Figure 1 shows the points of sampling, and Table II gives the results for the moisture determination.

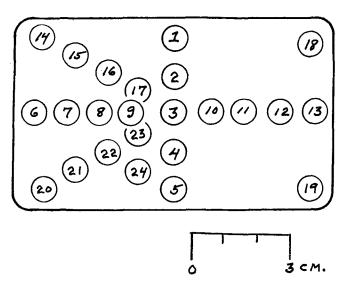


FIG. 1. Location of moisture-sampling sites in a single bar of soap.

TABLE II Moisture Content of a Bar Soan at Various Sites

Sample No.	Percentage moisture loss at 105°C.		
1	10.21		
2	10.91		
$ \begin{array}{c} 1 \\ 2 \\ 3 \\ 4 \\ 5 \\ 6 \\ 7 \\ 8 \\ 9 \\ 10 \\ \end{array} $	10,97		
4	10,45		
5	9.61		
6	9,39		
7	10.14		
8	10.48		
9	10.76		
10	10.75		
11	10.68		
12	10.10		
13	9.10		
$\frac{14}{15}$	8,95		
15	9.62		
16	10.34		
17	10.54		
18	9.12		
19	8.97		
20	8.88		
21	9.83		
22	10.13		
23	10.35		
24	9.99		

Inspection of the data in Table II shows that the moisture content of a bar varies over a wider range than is commonly suspected. The corners, in particular, dehydrate to a considerable extent and certainly cannot be ignored in attempting to determine the average moisture content of a bar product.

It will be noticed that the four corners of the bar, represented by samples numbered 14, 18, 19, and 20 yielded results varying from a low of 8.88 to a high of 9.12 as compared with the center of the bar (sample No. 3), which gave a moisture result of 10.97. The latter incidentally was the highest value for the 24 samples analyzed. Samples 14 to 17 progressed in magnitude linearly from 8.95 to 10.54 compared to the series 6 to 9 and 10 to 13 which varied from 9.39 to 10.76 and 9.10 to 10.75, respectively.

It would appear from both the viewpoint of accuracy and avoidance of possible disputes between laboratories that to obtain the correct average moisture content of a sample in the form of a bar, either the whole bar or a symmetrical portion, should be taken for analysis instead of slices from the interior of the bar.

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[Received August 12, 1957]

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Oils and Fats

Rheology and viscometry. S. Le Sota (National Lead Co., Philadelphia, Pa.). Paint and Varnish Production 47, 60(1957). The author gives a review of some of the fundamental concepts and definitions of rheology in outline form as an introduction to viscometry and continuous recording viscometry. A viscometer directory has been included to illustrate and describe the various types of viscometers, their units, range and prin-ciple of mechanism. The 34 figures illustrate the broad range of information provided.

Color measurement and control. O. H. Olson(Armour Res. Foundation, Chicago, Ill.). Paint and Varnish Production 47, 76(1957). The complexity of color and its dependence on many factors impede automation in color control. The author discusses the dimensional nature of color, the ingredients of color, instruments of color measurement, spectrophotometers, tristimulus colorimeters, and abridged spectrophotometers.

Deacidification with urea of ground-nut oils of low and average acidity. M. Loury. Olearia 11, 9/10, 213-217(1957). On the basis of previous experiments on the deacidification of oils by use of urea complexes the author suggests a plan for working directly on miscellas of the oil with solvent. The direct separation by centrifugal action of the deacidified oil, by the addition of a certain quantity of water, is possible. Although this method is more economical than ones previously described it still does not allow for a complete deacidification and needs to be further studied.

Soya seed and oil in the world. A. Ferrara. Olearia 11, 218-228(1957). After some introductory remarks on the importance of soya in the group of fluid vegetable oils, the author draws a general picture of the place of soya in the world, discussing the origin, spread, and characteristics of soya. He then goes on to examine soya products and their respective uses, the chief producing countries and the world trade in soya seeds and oil. At the end he describes the positions held by these products in 1951-55, the changes as compared to prewar years, and future trends.

A study of reversion of walnut oil. Mme. S. Durant-Veron and A. Prevot(ITERG). Rev. franc. corps gras 4, 502-505(1957). The authors discuss walnut oil produced in France, its reversion, causes and methods for detection. Two spectrum of the walnut oil with different stages of oxidation and examples with and without an antioxidant (Tioxan, 0.2% concentration) are shown.

Oxidation degradation in refining of edible oils. A. Dangoumau and H. Debruyne (Laboratoire Municipal de Bordeaux, France). Rev. franc. corps gras 4, 478-501(1957). The authors discuss the effects of oxidation as a degrading effect on certain edible fats and oils. Oleic and linoleic acid are considered in regard to peanut oil with 54 spectra given of the ultraviolet region from 2300 to 2900 millimicrons and one infrared spectrum \mathbf{shown}

The normal C_{17} fatty acids of musk-ox fat. Mary J. Chisholm and C. Y. Hopkins (Nat. Res. Council, Ottawa, Can.). Can. J. Chem: 35, 1434-1437(1957). A sample of body fat of the Canadian musk ox (Ovibos moschatus subsp.) was converted to methyl esters and distilled. The fraction containing esters of C17 acids was crystallized fractionally at low temperatures and two straight-chain C_{17} acids were isolated. *n*-Heptadecanoic acid was identified by analysis, by mixed melting point of the acid and two derivatives with authentic samples, and by its X-ray diffraction pattern. 9-Heptadecenoic acid was identified by analysis, by mixed melting point of two derivatives with authentic samples, and by oxidative cleavage. It is estimated that the fat contained 1.7% of *n*-heptadecanoic acid and 0.9%cis-9-heptadecenoic acid, based on the total fatty acids. Although there was an appreciable content of trans acids in the fat, the heptadecenoic acid was found to contain little or none of the trans form.

The properties of some vegetable oils. Seiichi Ueno and Kenji Matsushima (Kinki Univ., Fuse, Osaka). Abura Kagaku 6, 20-3(1957). The oil content and physical contents of oils extracted from the following seeds were studied: Stauntonia hexaphylla, Aralia chinensis, Kraunhia floribunda, Syringa amurensis var. japonica and Hibiscus manihot. The oil of the seed coat of Acorus calamus was also studied. (C. A. 51, 18654)

The course of hydrogenation of sardine oil. Maromi Takeda and Yoshiyuki Toyama (Nagoya Univ.). Abura Kagaku 5, 10-3 (1957). Purified sardine oil (acid number 0.5, saponification number 102.3, and iodine number 168.3) was hydrogenated in the presence of 2% nickel catalyst at 180° up to 8 hours.