Part III. Extraction Tests

See Table III for a comparison of the sieve analysis of the raw feed, and the expanded crumb; Table IV for the percolation data; Figure 4 for the plotted extraction curves. Although the expanded bran is compared directly to raw bran the work of Gastrock et al. (2,4,15) with filtration-extraction can also serve as a basis for comparison.

Discussion

Before a material can be expanded, it should be cooked. The cooking is obtained within the Expander barrel, and the expansion is obtained immediately upon discharge of the material through the die orifices. As a matter of procedure, pilot plant tests generally cover a range of cooking conditions to determine which conditions of cook are required to pretreat a material before expansion can occur. This was done with rice bran and the proper conditions are recorded in the data. Actually, there is a wide range of conditions adequate for the preparation of rice bran for solvent extraction through use of the Expander, perhaps wider than the range recorded. When the cooking conditions are relatively mild, the expanded product appears in an easily crumbled form. When the cooking conditions are more severe, the product occurs in a continuous rope-like form which can be cut into pieces of any desired length by means of a rotary knife blade attached so that it passes over the die orifices.

TABLE IV
Percolation Data

Sample	Penetration rate	Percolation rate	Drain time	M & V of marc
Expanded rope-like cake.....	241 in./min	140 gpm/ft^2	0.3 min	25%
Expanded crumb	98.4 in./min	35 gpm/ft^2	1.6 min	30%

The degree of expansion in both forms was minimal, which was to be expected, due to the low starch content in rice bran. The rope form is usually more desirable in an expanded product, but the crumb form was found to be superior to the rope form because it is more easily extracted in a solvent extractor.

The essential objectives in preparing rice bran for solvent extraction are 1) to prevent any rise in the FFA content; 2) to agglomerate the particles of bran together in order to reduce or eliminate the fines portion and to prevent channeling in the extractor baskets; and 3) to obtain a percolation rate in the extractor baskets of not less than 5 gpm/ft^2 in order to insure adequate mass transfer rates of oil from the bran into the solvent.

These objectives are fulfilled by expansion in both forms of finished product. But since the crumb requires a much shorter retention time for solvent extraction, due to its smaller particle size, it is preferred over the rope-like product. The retention time for the crumb is very smiliar to that required by flaked soybean. The extracted meal, moreover, is not as hydroscopic as extracted meal from raw rice bran; and the rice bran, after expansion and drying, can be stored under the usual conditions for long periods of time before it need be extracted for the rice oil .

ACKNOWLEDGMENTS

Assistance of G. A. Soltis in obtaining some of the extraction data; help from R. P. Gortz in obtaining the FFA data; aid from T. E. Pahoundis and Thomas Guilliford in the Expander operation; and aid from Jean Mehalik in preparing the manuscript.

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[Received April 28, 1964—Accepted October 22, 1964]

Determination of Weight of Bulk Oil Shipments

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Abstract

The following is a brief consideration of the methods used in the determination of wt of bulk shipments of vegetable and animal fats and oils, with particular reference to tallow. Sources of error are noted, and the techniques involved in accurate specific gravity determinations are discussed.

Introduction

As MOST BUYERS AND SELLERS of vegetable and animal fats and oils are aware, shipments by vessel are carried largely in bulk. Since buyer, seller, vessel owner and insurance agent all require accurate measurements or determinations of wt at both point of origin and point of discharge, we believe a brief discussion of the techniques involved would be of interest to the industry in general. This firm has been actively involved in the surveying, inspection and chemical work connected with bulk vegetable and animal oil shipments ever since the first delivery in bulk was made at San Francisco some 50 years ago. During this time we have witnessed vast changes in methods and practices and have had the opportunity of learning a great deal, not only from our own experiences, but from reports by other surveyors in many parts of the world. To the best of our knowledge, there is no universal code or system in use whereby all surveyors involved in any particular shipment can always intelligently interpret the findings of the others. With this in view, and with the hope of possibly aiding in the introduction of such a code or system, we would like to present briefly our procedures. Also during the presentation, we would like to point out to the uninitiated some of the pitfalls and difficulties that are encountered.

General Procedures

To determine the actual wt of a given parcel of oil or fat, delivered to, or discharged from a ship's deep tank, there are only three possible methods. These are actual weighing, gauging and metering. Each of these will be discussed, but with the greatest emphasis on gauging since this is the method most frequently used (and often abused).

Metering. Under ideal conditions of pump pressure and oil temp, metering is theoretically possible. However, ideal conditions are never met with, particularly during a discharge, and we do not believe metering is ever used effectively in the wt determination of bulk oil shipments. Further comments would therefore be of little value.

Actual weighing of the oil is by far the most desirable procedure, but in most cases facilities for such weighing are not available, or weighing is not expedient. Weighing can be, and is done through tank scales, by the use of tank cars, tank trucks, and even drums. Tank scales, where available and when properly constructed and used, probably yield the most reliable and quickest wt possible.

Tank trucks and tank cars can be used shipside for both loading and discharge wt, or in loading, for delivery to a sealed storage tank for later delivery to ship. Tank trucks (and cars) must be weighed full and empty over the same accurate platform or track scale. Stenciled tare wt should never be used. When a shipment is being loaded, truck or car should be weighed heavy before delivery to shipside, and light after emptying, in order that residual tare on each load is excluded from the total stated net wt. During discharges, weighing is reversed, the empty trucks or cars being weighed before use. If trucks are used, the point of weighing should be reasonably near to shipside, in order that the wt of fuel consumed by truck between ship and scale will not enter appreciably into the determination of the final total net wt.

Drum wts are feasible only for loading, and where labor is cheap and time of not too much importance. Here again, drums must be weighed heavy before emptying to ship, and light after emptying. Dry empty tares or average tares should not be used.

Gauging can be and often is quite accurate. However, in any gauge measurement, there are six factors involved, and an error in any one of these will create a discrepancy. Five of these factors are, or should be, under the direct control of the chemist or surveyor involved. These factors, each of which will be discussed in detail, are: 1) Accurate pre-calibration of the shore tank involved; 2) Proper sampling of the commodity in the shore tank in order that the sample used by the chemist shall accurately represent the commodity as a whole; 3) Accurate temp measurement of the commodity in the tank; 4) Accurate measurement of the depth (innage) or outage of the commodity in the tank; 5) Accurate specific gravity determination of the sample, at the temp of the commodity in the tank; and 6) Proper calculations based on the tank calibration and the data obtained in 3,4, and 5.

1. *Shore Tank Calibration.* The calibration of storage tanks is a procedure that has been practiced and perfected over many years. Manuals and texts covering this subject are readily available, and it is not the purpose of this paper to supplement the available information. Charts, when supplied, must be assumed to be correct unless proved otherwise. However, there are a few possible sources of error that could be mentioned. The bottoms of large steel tanks are not always absolutely rigid and may sag or buckle under varying loads of oil in the tank. Bottoms should be checked periodically with weighed oil or water to verify if depth of weighed materials agrees with tank chart under varying loads. If not, proper corrections must be calculated and applied to chart readings. Total depth at gauge point should be checked each time a gauge is made as a check against bottom movement.

The measurement of storage tanks for the purpose of constructing charts is usually performed at the prevailing atmospheric temp which would normally be between 60 and 90F. If tanks are used for tallow, which is often heated to 140F or more, the steel plates of the tank will expand with the increase in temp, and the actual volume in the tank at any given depth will be slightly greater than the chart indication. This increase in volume can be calculated readily from the known coefficient of expansion of steel. However, the error due to this source is very slight percentagewise and less than the limits of error in other factors to be discussed later. Further, this error is partially or wholly compensated for by the expansion of the steel tape used in measuring the apparent depth of the tallow. The larger the diam of the tank in relation to depth, the less the compensation will be.

Ship's tanks are usually calibrated for cargo measurements, but due to irregular shape and internal structure, charts can be only approx. Measurements are further complicated by the varying list and draft of the ship. It is our opinion that ship's tank measurements should never be used as a basis of outturn weights for settlement purposes.

2. *Sampling.* Needless to say, the sample taken from a storage tank to be used for analysis and for the determination of specific gravity must represent accurately the entire amt of the commodity in the tank. In the case of a dry liquid oil, such as refined cottonseed oil, this presents no problem. If moisture or solid stearines are present, these must be included accurately in their proper amt by sampling with a zone sampler (See AOCS Official Method C 1-47 and advertisement this Journal, Nov. 1964, p. 36.) and the sample should represent a complete core from bottom to top. When solid fractions are present, it is always preferable (but not always possible) to heat the contents of the tank sufficiently for complete liquification

TABLE I

Oil Pycnometer 0–2 (Tare Wt 34.503) High Temp

F	C	Water wt	Gallon water	F	C	Water wt	Gallon water
80	(26.67)	50.028	8.3085	110	(43.33)	49.764	8.2608
81	(27.22)	50.021	8.3072	111	(43.89)	49.754	8.2589
82	(27.78)	50.014	8.3059	112	(44.44)	49.743	8.2570
83	(28.33)	50.007	8.3046	113	(45.00)	49.733	8.2550
84	(28.89)	49.999	8.3033	114	(45.56)	49.722	8.2530
85	(29.44)	49.991	8.3020	115	(46.11)	49.710	8.2511
	(30.00)	49.983	8.3006	116	(46.67)	49.699	8.2491
and so forth to 109	(42.78)	49.774	8.2626	139	(59.44)	49.412	8.1990
				140	(60.00)	49.399	8.1966

before sampling and gauging. With commodities such as coconut oil, palm oil, and particularly tallow, preheating is absolutely essential and gauging temp must be sufficiently high for complete liquification. In the case of tallow, as will be discussed later under the subject of specific gravity, the commodity may be sufficiently liquid at temp between 100 and 115F to permit accurate sampling with a zone sampler. However, at these temp there always will be a varying amt of solid suspended fractions, depending somewhat on whether the tallow is cooling off or being melted. With tallow in this state, even though the sample accurately represents the tank content, it is impossible to obtain a specific gravity and wt/unit volume which will truly represent the physical state of the tallow in the tank at the time the sampling and gauging was performed. Consequently, if accurate gauges are to be made on tallow, temp must average over 120F, and no portion should be sufficently cool to permit crystallization.

3. *Temperature measurements* are generally taken at the time samples are drawn. Unfortunately, however, these measurements often are not given proper consideration. This is due partly to the fact that very few storage tanks provide facilities for adequate temp readings, as normally the only place readings can be made is through the manhole very close to the rim of the tank.

A cup thermometer may be used for temp readings or a thermometer may be placed in the oil in a zone sampler brought up from the level being sampled. Zone sampler must be allowed to remain at each level long enough to equalize temp. The number of readings needed to obtain a true average may vary greatly, depending upon the size and depth of the tank, and the thermal condition of the commodity in the tank. An absolute minimum, in our opinion, is three, namely top, middle and bottom during a core sampling. If these three readings do not agree within one or two degress, more closely spaced readings should be taken. With unheated liquid oils which have been at rest for a day or more, and where temp is reasonably close to atmospheric, the minimum readings are often sufficient. In the case of solidifying fats that require steam coil heating, or with any oil or fat that has been delivered to the storage tank at temp considerably above atmospheric, these minimum readings become entirely inadequate. In extreme cases it is often necessary to make readings at every foot during the core sampling, and if tank is equipped with openings other than the manhole, one or more further core readings should be taken.

To illustrate the necessity for extended readings, we have seen in the course of our gauging experiences, temp differences of as much as 50–60F between top and bottom of a storage tank. This is an extreme condition rarely encountered but it is a possibility as a result of pumping extremely hot oil overhead into a tank of cold oil. Some owners of storage tank installations appreciating the need for adequate facilities, have installed openings in tank tops properly spaced so that each opening is in the geometric center of that segment of the tank it is intended to represent. Generally four openings are adequate. With these facilities in large diam tanks it has been found that the average temp at one side of the tank can vary considerably from that at the other.

Between the extreme outlined above and a uniform tank at atmospheric temp, there are, of course, many variations. Consequently no set rule can be used, but the gauger must be prepared to tailor his operations to fit the conditions in order to insure that an accurate average temp is obtained. In a tank with a load of 400,000 gal, an error of only 1F will result in a weight difference of ca. 1300 lbs. Needless to say all thermometers should be checked for accuracy.

4. *Depth Measurements.* Accurate depth measurements can be made only if the tank is properly equipped for measuring and if proper techniques and equipment are used. Measurements always should be made at the same properly marked spot on manhole rim, and the point of contact on bottom of tank should be a smooth level steel plate. Total height between marked spot and bottom should be accurately measured and noted for each tank. This should be compared with similar measurements under varying loads in the tank to verify that tank bottom is rigid and unchanging under all loads. Measuring tape should be a high quality steel tape, free from kinks and bends and with clear readable figures. Bob on end of tape should be sharply pointed and of sufficient weight to keep the tape rigid at all times. Point of bob should be examined frequently to see if rough handling has blunted point and thus shortened the tape.

The actual reading for gauge purposes should be as follows: Using the known total depth, the tape and bob are lowered into the tank to within one-half in. or so of the bottom. Then with a fairly rapid motion, tape is lowered until bottom in just "felt" and quickly lifted to prevent the oil from creeping up the tape by capillary action. Tape is carefully rewound on reel until top of wetted portion is visible and readable. Reading is made of the oil depth and process is repeated after thoroughly wiping the tape for several inches below previous reading, until successive readings are identical. Reading should be recorded in notebook with oil mark visible and not left to memory for latter recording. Readings to the nearest one-eighth in. are generally acceptable but with proper care one-sixteenth in. measurements are possible. As a check against the depth (innage) in the tank it is always wise to make an outage measurement (distance from oil surface to marked place on manhole). Innage and outage must add up to total calibrated depth.

When gauging tallow at the high temp needed and with steam on in heating coils, the tallow often is found in motion. If motion is sufficient to move tape from vertical, accurate innage measurement is not possible. Under these conditions an outage measurement is essential. This subtracted from known total calibrated depth of tank will be the measurement to be used.

If commodity in tank is from discharge of a ship's tank or other sources, sufficient time (preferably 24 hr or more) must be allowed to permit all entrained air to dissipate and foam to clear. Should persistent foam remain, care must be taken to clear oil surface at point of gauge so that only clear liquid is measured.

5. *Specific Gravity.* The actual determination of specific gravity is, in our opinion, the most important step in a gauging operation, and in all probability the least understood. Hydrometer readings, with temp

TABLE II

Type of tallow	FFA %	Specific gravity at t/t			
		113F	122F	131F	140F
Fancy	2.60	0.90182	0.90038	0.89899	0.89779
Prime	5.90	0.90211	0.90050	0.89927	0.89799
Yellow grease	11.00	0.90582	0.90443	0.90307	0.90184

corrections, while used extensively in the petroleum industry, should never be used in gauging for the vegetable and animal fat trade.

The most accurate and convenient instrument for such a determination is a glass pycnometer, and the determination should be made at the same temp as the commodity in the tank. A 50-ml pycnometer is, in our opinion, the most convenient. Those carried in stock usually are equipped with a standard taper thermometer with a temp range of from 14–36C (57F–97F). This generally is sufficient for liquid oils but for solidifying fats—tallow in particular—special thermometers reading up to 60C (140F) or more can be made to order. Thermometers must be accurate and the scale should be calibrated in 0.2 divisions. We find a C scale more convenient and F readings in tanks are readily convertible into C. Tare wt of the empty pycnometer is carefully determined on an analytical balance to the nearest mg. If numerous determinations at varying temp are to be made, it is exceedingly helpful to construct a table for each pycnometer showing each °F with corresponding °C, water wt, and pounds of water/gal. A portion of such a table is shown in Table I. Pure distilled and recently boiled water should be used in the calibration. Actual calibrations with water should be made at the maximum and minimum temp on the thermometer and at numerous closely spaced points between. These can be plotted on a large scale graph and the values for each degree read off. The graph for water is not a straight line.

The actual determination, both with water for the calibration and with the oil or fat for each gauge, is as follows: Pycnometer is filled with the well mixed sample and held at a temp slightly lower than the determination requirement until all entrained air has risen into the neck. The air bubbles are displaced with additional sample. Thermometer is then carefully inserted and firmly seated. Pycnometer is placed in a water bath held at a temp not more than 0.3° above the determination temp. Contents are gradually heated to the desired point while pycnometer is constantly rotated by hand, using the portion of the thermometer extending above the surface of the water. As the contents are heated and expanded, excess sample is expelled through the side arm. When the desired temp is reached, tip of side arm is wiped off, and side arm cap replaced. Pycnometer is then carefully wiped off and dried and weighed on the analytical balance to the nearest mg. Process is repeated until successive weighings agree within 2 mg or less. With careful manipulation, duplicate determinations are often identical. The use of a water bath as a means of raising the pycnometer contents to the desired point is absolutely essential. Without its controlled heat, outer portion of contents can easily be overheated due to the relatively high viscosity and slow heat transfer.

Tare wt is subtracted from the gross, giving the net wt of the sample in the pycnometer at the desired temp. Water wt at the same temp is determined in the same manner, or read off from the previously prepared table. Sample net wt divided by the water wt at the same temp gives the specific gravity in air at t/t. Calculation is carried to the fifth decimal. This multiplied by the wt of a gal (cu ft or liter) of pure water at t° gives the wt of one gal (cu ft or liter) of the sample for use in the final calculations. Water wts/unit volume are obtained from Table 33 for gal and Table 34 for cu ft in Circular No. 19 of U.S. Bureau of Standards. Wts in air and not in vaccum are used. In this connection it should be noted that the values listed in Tables 31 and 32 for density of water should not be used. These represent densities and not specific gravities and will not give correct wts if used with specific gravities determined in air. To convert a water wt in lb/gal in air (Table 33) to kg/liter, a factor of 0.119829 should be used.

Tare wt of pycnometer should be checked frequently to correct for any losses by chipping or abrasion. If table is prepared and used, water wt should be checked frequently at different points. With repeated use the standard taper thermometer can be ground slightly deeper into the neck of the pycnometer permitting the lower portion to displace a greater amt of the liquid, thus giving lower wt. During the actual determination in the water bath, with the pycnometer in constant rotation, care must be taken to prevent wear by abrasion of the glass on the bottom of the bath. It should be noted here that the table prepared and illustrated above is valid only for one pycnometer. It is essential therefore that the operator take particular care in handling to avoid breakage.

When a determination of the specific gravity at the actual temp of the gauge is made, the use of average expansion factors is avoided. Different samples of the same type of oil or fat are similar but not necessarily average. Further, the use of an average specific gravity should never be substituted for an actual determination if accurate results are desired. To illustrate the variation in one type of commodity, tallow, the following determinations on three types at varying temperatures are listed below.

The differences shown are very significant. Further, these specific gravities also can be affected by the varying amt of moisture and insolubles present in the sample.

As a further illustration concerning the necessity of having the temp in a storage tank of tallow sufficiently high for complete liquification, we determined the specific gravity twice on the same sample at 104F. The first test was made immediately after sample had been completely melted and quickly cooled without any crystallization, and the second somewhat later after crystallization had progressed to a marked degree. Under these conditions the successive weighings did not agree. The two results on the same extra fancy tallow, both at 104/104F were 0.90328 and 0.90505. Corresponding lb/gal were 7.4703 and 7.4849. The difference between these (0.0146 lb/gal) could be a serious factor in a gauging operation.

6. *Calculations and Report.* The final report on any gauge used for trading purposes should be clear, concise and readily understood by any interested party. While the exact form of the report might vary, it should include the following: a) A statement of the depth of the commodity being gauged at the average temp (t) existing in the tank at time gauge is taken; b) Equivalent gal (liter or cu ft) based on accurate chart; c) Specific gravity of the commodity at t/t carried to five significant figures; d) Wt (in air) of one gal (liter or cu ft) of water based on a reliable source such as the U.S. Bureau of Standards Circular No. 19. e) Wt of one gal (liter or cu ft) of the commodity in the tank, based on values (c) and (d); and f) Finally the wt in lb (or kg) based on the values recorded in (b) and (e).

Such a report is self explanatory and can be read and utilized or criticized intelligently by any interested party.

[Received July 8, 1964—Accepted September 9, 1964]