THE ANALYSIS OF TUNG FRUIT*

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Abstract

Methods applicable to the routine analysis of tung fruits were developed by taking into account such factors as variation in moisture content of individual fruit, high oil content of the tung kernels and the high reactivity of the extracted oil. It was found that in a closed container the moisture distributes itself evenly between individual fruits, that the oil can be extracted completely from tung kernels if they are reground with sand, and that the moisture can be removed from ground tung kernels and the solvent completely removed from the extracted tung oil without deterioration in a vacuum oven at 100° C. with 28-29 inches vacuum.

THE increasing importance of the Tung Oil Industry in the South with five modern tung oil mills operating and two more being erected in the tung oil centers has made it seem worth-while to describe the analytical methods that have been developed in this laboratory. Effort has been made to develop procedures that are rapid and applicable to routine work, and yet capable of yielding reliable and accurate results.

The wide variations in the moisture content of the different fruits in the samples and in the moisture content of the hull, shell and kernels of the individual fruit was found to be a disturbing factor in connection with the analysis of tung fruits. However, it was found that if the sample of tung fruits is kept in an air tight can for a week, the moisture will distribute itself evenly throughout the sample. For example, for ten individual fruits, the standard deviation for moisture content was 0.11% and 0.465% with two samples of tung fruit. If a ten-fruit sample is selected, the standard deviation between samples for moisture content would be 0.035% and 0.15%, which amount would not be significant in the analysis.

The high oil content of the tung fruit kernels and the high reactivity of tung oil are factors which must be taken into account. When tung kernels were ground or rolled fine enough to permit the removal of the oil by a single extraction, low results were obtained, indicating that oil was lost in the preparation process. It was found that a vacuum oven must be used in the removal of moisture from the ground kernels and in the removal of the last traces of solvent from the oil to prevent oxidation changes which seriously affect the moisture data and the quality of the extracted oil. The removal of traces of moisture from the extracted oil in the vacuum oven was facilitated by adding about a half cubic centimeter of acetone to samples containing appreciable amounts of moisture before placing the flask in the vacuum oven.

THE METHODS IN DETAIL

Laboratory Sample. — The analyst should examine the sample at the laboratory and clean it, if necessary, of foreign matter and report as such. A sample of about 80 fruits is placed in a two gallon air-tight container (lard can) for one week to allow even moisture distribution.

Sampling and Separation into Component Parts — The cleaned fruits are spread out upon a table and two subsamples of ten fruits each are selected at random by the chemist for the analyses. The selected sample is carefully separated into its component parts (hulls, shells and kernels). The three separated components are weighed and the percentage of each calculated. The total quantity of the component parts, as determined, is taken to be the quantity of the fruit sample. Thereby, any moisture changes in the fruits during the separation of their component parts are eliminated from consideration.

Moisture Determination. — Approximately 20-gram portions of the separated hulls, shells, and undamaged whole kernels are weighed into tared aluminum dishes (1″ high and 4″ diameter) and the dishes are placed in a drying oven at 101° C. to 102° C. until dry. A 24 hour period was found to be sufficient. The dishes are removed from the oven and placed in an efficient desiccator until cool (one-half hour) and weighed, the loss in weight being considered as moisture. The moisture in the fruit sample is calculated from the determined moisture content of its component parts.

Oil Determination. — The remaining undried tung kernels are ground twice in a Russwin food grinder, No. 1 type, using the sixteen tooth cutter. An evaporating dish of appropriate size is used to collect the ground sample which is then thoroughly mixed by shaking in a wide mouth jar. Two 5-gram portions of the ground kernels are weighed accurately and are spread in a thin layer on a 150 mm filter paper (Reeve-Angel No. 211 or equivalent). The paper is folded over the sample toward the center on two parallel chords at points about one-quarter of the diameter distant from the center. The paper is then wrapped by coiling, starting at one end of the paralled diameter, into a cylinder. The sample is then wrapped in a second paper in such a manner as to prevent escape of the meal, leaving the top open like a thimble. A piece of cotton is placed in the top of the thimble to distribute the dropping solvent. Several turns of white cotton thread (No. 30) is used to keep the papers from unwrapping. The thimble is placed in a Butt extraction tube, which is attached to a tared 150 cc. flat bottom wide neck extraction flask containing about 45 cc. of petroleum ether (boiling range 35° to 60° C., and meeting the American Oil Chemists' Society's specifications); the tube is connected to a water cooled, 400 mm. Allihn bulb condenser; and the sample is extracted over a steam bath for 4 hours.

The sample is then transferred to a mortar and ground vigorously, first alone and then with one gram of fine sand (Merck & Co., No.

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33018) for 5 minutes. The sample is then rewrapped in the filter papers and re-extracted over the same flask for an additional 2 hours.

The major portion of the inflammable solvent vapors is removed carefully on the steam bath, using distillate collectors similar to Pyrex 3320 without ground joints, ten to fifteen minutes being sufficient. Prolonged heat at atmospheric pressures causes an appreciable oxidation of the oil. The remaining solvent is removed by placing the flask in an oven at 100° — 102° C. under reduced pressure. The lowering of the pressure in the vacuum oven must be done slowly and cautiously to prevent violent bumping in the flasks and loss of the determinations. The flasks are placed in the oven for 3 to 5 minutes to heat to oven temperature and the pressure is lowered in steps of 5 inches, allowing 2 minutes between steps, until the full capacity of the oven (28-29 inches) is reached. Complete removal of the solvent in the oven was accomplished in 40 minutes at 28-29 inches. The flasks containing the oil extract are cooled to room temperature, weighed and the percentage of oil in the tung fruit kernels calculated.

Iodine Number Determination. — The iodine number of the extracted tung oil is determined by the Wijs Method, as given in the "Methods of Analysis of the Association of Official Agricultural Chemists," 4th Edition, 1935, p. 412, with the following modifications:—

175 mg. ± 2 mg. of the tung oil sample is taken for each analysis and the temperature of the reacting solution is maintained at 20° C. for the 30 minutes allowed. In the preparation of the Wijs solution, glacial acetic acid must be used that passes the permanganate test (Murray's Standards and Tests for Reagents and C. P. Chemicals, 2nd Edition, 1927, p. 7). Wijs solutions prepared with inferior grades of acetic acid deteriorate rapidly and yield incorrect, low iodine values.

Example of Calculations

Weight in Grams		Percentage
321.0	Fruit	100.0
160.4	Hulls	50.0
58.0	Shells	18.1
102.7	Kernels	31.9
	Moisture in Hulls	17.5
	Moisture in Shells	10.2
	Moisture in Kernels	7.2

Moisture in Fruit = 17.5 x 0.50 + 10.2 x 0.181 + 7.2 x 0.319 = 12.90%

Oil from 5 g. kernels (3.1948g = (61.83%
(3.1740g (61.93%

Oil in Dry Kernels = 61.88 ÷ .928 = 66.68%

Oil in Wet Fruits = 61.88 x 0.319 = 19.74%

Oil in Dry Fruits = 19.74 ÷ 0.871 = 22.66%

Iodine Number (Wijs)	I. No.
Wt. Sample 0.1747 g.	163.0
Wt. Sample 0.1749 g.	163.4

Report of the Committee on Petroleum Ether Specifications

OWING to the fact that petroleum ether having objectionable characteristics had been supplied to some members, which petroleum ether complied with the American Oil Chemists' Society's specificatons, a request was made for a revision of the specifications which would eliminate the undesirable characteristics. The specific points to be considered were odor, saponification value, and mineral acid residue. In view of the fact that the solvent power of petroleum ether is dependent upon the composition of the hydrocarbons present, the committee thought it advisable to narrow the distillation requirements so as to maintain the solvent power of the petroleum ether in a more narrow range. This was accomplished by adopting as standard the distillation characteristics of the petroleum ether which has been used by the major portion of the cottonseed crushing industry. Since the purchase of cottonseed on analysis is in effect in some portions of the country, the desirability of fixing the solvent effect of the petroleum ether is evident. The following specifications for petroleum ether are recommended to replace the present specifications:

PETROLEUM ETHER SPECIFICATION

Initial boiling temperature	Not less than 35°C
Initial boiling temperature	Not over 38°C
Dry flask end point	Not over 60°C
Dry flask end point	Not less than 52°C
At least 95% distilling under 54°C.	
Not over 60% distilling under 40°C.	
Specific gravity at 60°F	0.630 to 0.660
Color	Water White
Evaporation residue 100 cc	Not over .0011 grams
Doctor Test	Sweet
Copper Strip Corrosion Test	Non-corrosive
Unsaturated compounds	Trace only permitted
Residue in Distilling Flask	Neutral to methyl orange
Blotter Strip Odor Test	Odorless within 12 minutes
Aromatic Compounds	No nitro benzene odor
Saponification Value	Less than 1.0 mg. KOH per 100 cc.

Distillation test to be made according to A. S. T. M. D 216-32. As a check on the evaporation residue, 250 cc of the petroleum ether and 0.25 g. of stearin or other hard fat (previously brought to constant weight by heating) when dried as in the actual determination shall not show an increase in weight exceeding 0.003 g. Copper strip corrosion test is made by inserting a small polished copper strip into the petroleum ether in the distilling flask. There should be no appreciable darkening of the copper.

Unsaturated compounds shall be determined by the method for determining olefins on page 154 of the March 15, 1938 Analytical Edition of Industrial and Engineering Chemistry. Odor Test: Immerse one inch of a strip of white, unglazed blotting paper, approximately 1" x 4" x 0.166" in size, in the petroleum ether for 30 seconds, remove strip and allow to dry at room temperature in still air for 12 minutes. Aromatic Compounds: Add 5 drops of petroleum ether to 40 drops of concentrated sulphuric acid and 10 drops of concentrated nitric acid in a test tube, warm for 10 minutes, allow to cool for one-half hour, transfer to a shallow dish and dilute with water.

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