

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 react-

ing 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 (3.1748g)	(61.83%
	(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 specifications, 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 de-

termining olefins on page 154 of the March 15, 1938 Analytical Edi-

tion 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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