Rapid Method for the Preparation of Samples and Evaluation of Oil Quality in Small Samples of Flaxseed[1*]

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ONE of the major problems confronting the originators of new varieties of flaxseed is the application of suitable quality tests to large numbers of small samples, permitting the selection of promising strains and the discarding of unsatisfactory lines early in the breeding program. The oil content of flaxseed is positively correlated (1, 2, 3) with seed size, but a measure of the quality of the oil requires a determination of its iodine value. Since this information is frequently required for the seed from single plants, which may not yield more than three grams, special methods applicable to such small samples which can be rapidly conducted with large numbers of samples are necessary.

Originally this problem was met in the flaxseed laboratory at the Minnesota Agricultural Experiment Station by refractometric estimation of the iodine value of the oil obtained by partial extraction of small samples with petroleum ether. The samples were ground by hand with sand in a glass mortar, transferred to filters, and extracted several times with small portions of petroleum ether. The filtrates, collected in 50-ml. Erlenmeyer flasks, were evaporated on a steam bath, dried for 30 minutes at 100°C. in a vacuum oven, and the iodine values of the oils were determined refractometrically. This last step is more fully described below.

Subsequently a much less laborious and more rapid procedure involving the use of an Intermediate Wiley Mill was developed. As the techniques for preparing and analyzing the extracts may prove useful to others, they are presented here.

Flaxseed Sample Grinding and Oil Extraction

Samples are handled in sets of 24 throughout and 50-ml. Erlenmeyer flasks are numbered in advance for several of these sets. Likewise, several sets of filters are folded in advance so that the grinding may proceed without interruption.

The first set of flasks is provided with 50-mm. funnels and 9-cm. filters held in place by paper clips. The Intermediate Wiley Mill is operated with a 20-mesh sieve and runs continuously during a set. A flask with funnel and filter is placed under the delivery spout of the mill, and approximately one gram of flaxseed is poured into the hopper. The wooden plug in the hopper is momentarily lifted to drop the seed into the grinding chamber. In less than a minute the sample is completely ground and falls upon the filter. A wash bottle equipped with a rubber pressure bulb and pinch cock is used to direct a stream of petroleum ether (Skellysolve F) into the hopper and grinding chamber. After draining a few seconds, the chamber is washed with a second small portion of petroleum ether. The front glass plate is then removed from the mill and the grinding chamber, sieve and delivery spout washed down into the filter with petroleum ether for a total of about 15 ml. The glass front plate is wiped with a towel and replaced, the flask and filter removed to drain, and another substituted for the next sample.

At the completion of a set the flasks are placed upon a steam bath, evaporated, dried 30 minutes in a vacuum at 100°C., and cooled, after which the oils are ready for iodine value estimation. While the solvent is evaporating from this set, the used filters are removed from the funnels which are then rinsed by dipping them into a beaker of petroleum ether. New filters are inserted and the funnels are placed ready in the next set of flasks. In this manner, grinding, evaporating, drying, and estimating iodine value may proceed simultaneously if a second technician is available. Otherwise, iodine values may be determined alternately with the preparation of several sets of oils.

Refractometric Estimation of Iodine Value

Iodine values are at present derived from scale readings of the oils at 25°C. in a Zeiss double heating prism refractometer. These readings are directly converted to iodine values by referring to a chart computed from the relation between refractive index and iodine value as reported by Zeleny and Coleman (4). If a refractometer is not at hand, Wijs iodine values of the oils may be determined rapidly as follows:

Micropipetting Technique for Wijs Iodine Value Determination

A small quantity of oil, free of bubbles, is taken by means of an ordinary rubber-bulb, dropping pipette and transferred to a 0.1 ml. Hagedorn-Jensen precision blood-sugar pipette by capillarity. To do this both pipettes are held horizontally, tip to tip, and are separated immediately when the precision pipette has been filled exactly to the mark. If one goes beyond the mark the pipette must be washed out with carbon tetrachloride and dried by aspiration and the procedure repeated. The tip of the precision pipette is wiped off with clean cotton, care being taken not to touch the orifice.

The average weight of the oil sample is obtained by weighing the pipette before and after filling several times; with a little practice the weight of oil does not vary more than ± 0.1 mg.

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The contents of the pipette are washed directly into an iodine value flask by connecting it to a dispensing burette by means of a short piece of neoprene tubing and delivering 10 ml. of carbon tetrachloride through it. The pipette is then dried by aspiration, when it is ready for the next oil sample.

The remainder of the method follows the usual procedure for the Wijs iodine value determination except for the Hoffman and Green (5) modification employing 250 mg. of mercuric acetate as a catalyst which permits titration within three minutes. With two pipettes one may be dried while the other is in use. Care must be taken, however, in noting oil weights if the pipettes are not exact duplicates. The dropping pipette is cleaned by removing the rubber bulb, rinsing in carbon tetrachloride and drying by aspiration.

Following are a few typical results using the above technique:

Sample	Wt. Oil g.	Iodine Value	
		Wijs	Refractometric
1	.0915 .0916	176.0 176.6	175.6 175.9
2	.0914 .0914	161.2 161.2	161.2 160.7
3	.0914 .0914	161.7 161.8	161.5 161.5

Discussion

The point may be raised as to the safety of introducing highly inflammable petroleum ether into the moving Wiley mill. These mills are equipped with induction motors so that sparking occurs only upon starting or stopping. Consequently there is less hazard in continuous, than intermittent, operation, even if the few ml. of solvent involved could be considered a hazard. The present mill has been used for two years, grinding several thousand samples without an incident.

Insufficient data have been obtained on pipetting technique as to the possible range of oil weights due to differences in specific gravity. However, Jamieson (6) gives extreme figures for raw oil of .931 and .938, which would be reflected in a possible deviation of approximately 0.7 iodine value from the mean if the oil samples are pipetted without checking the weight. Of course, the exact weight of oil can be readily obtained by weighing the pipette before and after filling if greater accuracy is desired.

Summary

1. One-gram samples of flaxseed, such as are obtained from single plants in breeding work, may be prepared rapidly in quantity for iodine value estimation of oil quality by grinding in an intermediate Wiley mill and partially extracting immediately on a filter with petroleum ether.

2. The oils obtained after evaporating and drying the extract are sufficient in quantity to permit the determination of iodine value either refractometrically or by a modified Wijs method in which the oil sample is measured by a precision pipette involving a special technique.

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Abstracts

Oils and Fats

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