seem to inhibit drying properties less than do residual hydroxyls, and the drying oils obtained from nearly completely split oil-fatty acid esters seem to show some superiority as compared to catalytically dehydrated and thermally bodied oils of comparable, iodine number, viscosity, and acid number. This conclusion however need not be general since the drying properties are sensitive to other variables. For example, as indicated by process product 1, prolonged processing at high temperatures can improve drying rates even though the conditions are insufficient to cause much change in secondary ester content, iodine value, or diene value. Possibly other mechanisms, such as ester exchange and other isomerization reactions, are important, the effect of which cannot be detected by the methods of analysis here employed.

The differences in drying rates of dehydrated castor oils of this process and other processes are minimized greatly by the introduction of metallic drier catalysts hence, from the viewpoint of comparing the commercial worth of dehydrated castor oils, the quality of drying rate differences may not be very important. Dehydrated castor oil is seldom used as the sole nonvolatile component of the binder in paint vehicles, and hardly ever in any form without the aid of drying catalysts.

Apart from the subject of drying rate comparisons for different process oils, the data of this paper should have considerable qualitative significance in the formulation of other vehicles where castor oil products are used to modify polyesters or other varnish resins. The esterification possibilities of secondary hydroxyls of ricinoleic acid and the extent of thermal stability of such esters in the presence of weak acids are facts important to the science and art of the resin formulator.

Acknowledgment

Appreciation is expressed to the Sherwin-Williams Company of Cleveland, Ohio, who generously provided the materials and equipment for this experimental study. The yet unpublished analytical method for determination of ester-split value is credited to the research laboratory of the Sherwin-Williams Company.

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[Received December 3, 1953]

Report of the Seed and Meal Analysis Committee, 1953-1954

THE work of the Seed and Meal Analysis Committee is conducted by eight subcommittees. They are: Screen Test for Soyflour, Analysis of Tung Fruit and Meal, Analysis of Copra and Copra Meal, Analysis of Castor Beans and Pomace, Foreign Matter in Lint, Residual Lint on Cottonseed, Analysis of Flaxseed and Linseed Meals, and Bulk Sampling of Meals. Development of methods for the analysis of sesame seed has been requested of the committee. A subcommittee will be organized for the work on such methods as soon as success in the development of an instrument by which the seed can be ground in a solvent is assured. It appears that the subcommittee may be named soon. Reports and recommendations of two subcommittees are as follows:

Analysis of Flaxseed and Linseed Meal

METHODS for sampling and for the determination of moisture and volatile matter and of oil in flaxseed, and of moisture and volatile matter, oil, protein, ash, and crude fiber in linseed meal have been studied collaboratively by the subcommittee. Methods proposed are based on the collaborative tests and the judgment of the members of the subcommittee and others experienced in the sampling and analysis of both flaxseed and linseed meal. The one for sampling flaxseed is essentially the federal procedure written in A.O.C.S. format. Those for the flaxseed are as follows:

Sampling

Scope: Applicable to flaxseed.

A. Apparatus

1. Trier, double-tube, separate compartment, grain trier or probe *ca.* 60 in. long.
2. Bag trier.
3. Sampling canvas 5 ft. x 2 ft.
4. Boerner sample divider, 36-pocket size.
5. Water-proof sample sacks.
6. Air-tight containers for preserving samples.
7. Spout sampler or "Pelican" for sampling bulk grain from running streams.
8. Carter dockage tester for flaxseed containing a No. 000 riddle, No. 1 riddle, a top No. 4 slotted sieve (0.064 x 3/8 in.) in the middle, a No. 2 round hole sieve (5/64 in. dia.), and a No. 7 round hole sieve (4 1/2/64 in. dia.) in the bottom carriage.
9. Hand sieves 0.064 by 0.375 in. perforations, 5/64 by 3/8 in. perforations, 1/2 in. round hole, 4 1/2/64 in. round hole, and 4 x 16 mesh to sq. in.

B. Procedure

a) *Flaxseed in bulk*

1. Probe in seven or more places with a standard trier in well distributed parts of the car. Insert the probe at an angle of 10 degrees with the vertical, with the slots closed and faced up. Open the slots and give the probe several gentle up and down movements. Then close the slots and withdraw the probe.
   a. Probe in center of car.
   b. Probe 3 to 5 ft. back from door-post toward the end of the car and approximately 2 ft. out from one side of the car.
   c. Probe from 3 to 5 ft. from same end of the car and approximately 2 ft. from opposite side of car, as described in b.
   d. Repeat b and c above in opposite end and sides of car.
   e. Probe 2 ft. from each end of car in center.
2. Place contents of each probe on the sampling cloth and mix by lifting alternate edges of the cloth. A minimum sample of 2,500 g. shall be collected and stored in an air-tight container.

b) *Flaxseed in truck loads or small bins*

1. Use same procedure as for flaxseed in bulk.

c) *Flaxseed in bags*

1. Take samples from the bags by any standard or approved grain trier of sufficient length to reach center of bag.
2. Draw samples from as many individual sacks, selected at random through the lot, as will be necessary to obtain a representative sample of at least 2,500 g.

d) *Unloading and loading flaxseed*

1. Where a sample is to be taken during loading or unloading operation, a pelican sampler device is used. Samples are taken at regular and frequent intervals to assure a correct and representative sample.

C. Cleaning and Reduction of Sample

1. Reduce sample size to *ca.* 1,000 g. by Boerner sampler.
2. Pass the reduced sample through the Carter dockage tester once.
3. Use the 000 riddle. If this should clog or kick whole flaxseed kernels over, use the No. 1 riddle.
4. When matted lumps of flaxseed clog the riddle or kick over with the coarse dockage, remove the riddle and top sieve No. 4 and proceed to make the determination provided above. In hand-picking a portion of the mechanically cleaned flaxseed to complete dockage determination, the matted kernels of flaxseed, even though portions of flax bolls adhere to them, shall be considered as flaxseed.
5. Examine the material that passed over the top sieve (No. 4), and if material contains large-sized flaxseed, it shall be rescreened with a hand sieve having 0.064 by 0.375 in. perforations, and if it contains small-sized flaxseed, rescreen with hand sieve having 3/64 by 3/8 in. perforations, recovering the flaxseed left in this portion.
6. If material that passed over the bottom sieve (No. 7) contains considerable pigeon grass seed or mustard seed, it shall be rescreened with hand sieve having 3/64 by 3/8 in. perforations. The material remaining on top of this sieve shall be considered as dockage. The material which passed through the sieve shall be returned to the mechanically cleaned sample.
7. If the flaxseed (mechanically cleaned sample) contains excessive amounts of pigeon grass seed and/or other weed seeds of similar size and shape, place *ca.* 250-g. on a hand sieve having 3/64 by 3/8 in. perforations, work the sample back and forth lengthwise of the perforations until all of the removable material has passed through the sieve. Repeat the operation until the entire sample has been sieved.
8. The flaxseed in the material that has remained on top of the hand sieve shall be reclaimed in the following manner: Use the hand sieve having 1/12 in. round hole perforations (fine-seed sieve), hold the sieve at an angle of from 10 to 20°, place the material on the lower edge of the sieve and strike the lower edge of the sieve with one hand in such a manner as to cause the material to bounce up and down. This will cause the pigeon grass, etc., to pass through the perforations of the sieve. Continue the operation until all of the separable dockage material has passed through the sieve. The material which remains on top of the sieve shall be returned to the cleaned flaxseed. If the material which passes through the sieve consists of 50% or more of whole or broken kernels of flaxseed, it shall be put back in the cleaned flaxseed, otherwise it shall be added to the dockage.
9. The mechanically cleaned flaxseed including the flaxseed reclaimed by hand sieving shall be run through the Boerner divider. Analyze a sample of at least 15 g. by hand-picking to determine the remaining dockage.
10. Store the cleaned flaxseed sample in an air-tight container.

D. Calculations

Calculate the foreign matter as follows:

Foreign matter in the original sample, % =

$$FM + [(100 - FM) \times fm/100]$$

where FM is % of foreign matter in large sample, fm is % of foreign matter in hand-picked sample.

Moisture and Volatile Matter

Definition: This method determines the moisture and any material which is volatile under the conditions of the test.

Scope: Applicable to flaxseed.

A. Apparatus

1. Forced draft oven, A.O.C.S. Specification H 1-39.
2. Aluminum moisture dishes, 30-gauge, 2 x 3/4 in. (*ca.* 50 x 19 mm.) with tight fitting slip-over covers.
3. Desiccator containing an efficient desiccant. See A.O.C.S. Specification H 9-45.
4. Boerner sampler or divider, 36-pocket size.
5. Air-tight sample containers of convenient size.

B. Preparation of Sample

1. Reduce the mechanically cleaned sample (Sampling Section C, paragraph 10) through the Boerner sampler to *ca.* 100 g. Preserve the sample in air-tight container and use on all subsequent analyses.

C. Procedures

1. Weigh *ca.* 10 g. of whole flaxseed into a tared moisture dish.
2. Dry in the forced draft oven at 130° ± 3°C. for 3 hours. Remove from the oven, cover immediately, cool in a desiccator to room temperature and weigh.

D. Calculation

$$\text{Moisture and volatile matter, \%} = \frac{\text{Loss in weight} \times 100}{\text{wt. of sample}}$$

Oil Content

Definition: This method determines the substances extracted by petroleum ether under the conditions of the test.

Scope: Applicable to flaxseed.

A. Apparatus

1. Butt-type extraction apparatus, assembled as indicated in the illustration (A.O.C.S. Official Method Aa 4-38).
2. Filter paper, S. & S. No. 597, Reeve-Angel No. 211, Whatman No. 2, or equivalent, 150 mm.
3. Absorbent cotton, free of petroleum ether extract.
4. Porcelain mortar and pestle. The mortar must be at least 4 in. i.d. at the top. The pestle handle must be large enough to afford a firm hand grip.
5. Roller mill, Allis Chalmers 6 x 6 in. With cast iron frame, metal feeder housing, bronze bushings, rolls corrugated 28-sawtooth with 1 in. spiral per foot and running sharp to sharp including flat belt pulleys to drive at 2:1 differential from a line shaft below machine.
6. Sieve, Tyler No. 20 (U. S. No. 20).
7. Brushes
   a. Round, 1-in. bristle.
   b. Steel brush, 6-in.
8. Flaxseed dockage sieve, wire mesh, 4 x 16 meshes per sq. in.
9. Aluminum moisture dishes, 30-gauge, 2 x 3/4 in. (*ca.* 50 x 19 mm.) with tight fitting slip-over covers.

B. Reagents

1. Petroleum ether, A.O.C.S. Specification H 2-41.
2. Sea sand, 30-mesh, washed and ignited.

C. Preparation of Sample

1. Quarter sample (Moisture and Volatile Matter, Section B, paragraph 1) until approximately 25 g. remains. Hand-pick to free of all foreign matter.
2. Pass the sample through the mill with the rolls set loosely so that the seed is thoroughly crushed. Tighten rolls and pass the crushed seed through the mill. Loosely set rolls give coarse material having poor extractability, and too tight a mill setting gives a mushy sample. The ground sample should pass through the No. 20 sieve (except perhaps a few hulls). If a sieve analysis is made to check grind, brush the sample lightly through the sieve, using the 1-in. bristle brush. Do not shake. Thoroughly clean

the rolls with the wire brush after each grind, and wipe the scrapers with a cloth.

3. Place the entire ground sample, including the material cleaned from the rolls, onto the 4 x 16 wire mesh sieve and brush lightly through the sieve, using the 1-in. bristle brush in order to break up agglomerate tending to destroy uniformity.
4. Mix the sample thoroughly by rolling the ground screened sample back and forth on glazed paper. Transfer to an air-tight container. This sample is used for the determination of oil, nitrogen, and second (ground) moisture. Weighings for all determinations should be made at the same time.

D. Procedure

1. Weigh accurately 3 g. of the ground sample into a filter paper and enclose in a second filter paper folded in such a fashion as to prevent escape of meal. (See illustration in A.O.C.S. Official Method Aa 4-38.) The second paper is left open at the top like a thimble, and a piece of absorbent cotton may be placed in the top of the thimble to distribute the solvent as it drops on sample.
2. Place wrapped sample in the Butt extraction tube and assemble the apparatus as shown in A.O.C.S. Official Method Aa 4-38. Put *ca.* 25 ml. of petroleum ether into a tared extraction flask before attaching to tube.
3. Heat on water bath or electric hot plate at such a rate that the solvent will drop from condenser on the center of the thimble at the rate of at least 150 drops per minute. Extract for 2 hours.
4. Remove thimble from Butt tube, allow petroleum ether to evaporate at room temperature. Transfer material in thimble to a mortar, add *ca.* 10% by volume of sand, and grind. Grind for one minute or with 100 vigorous strokes.
5. Return the reground sample to the same filter paper and continue the extraction for 3 hours. Add additional petroleum ether to provide for some loss due to evaporation.
6. Cool and disconnect the flask. Evaporate the solvent on steam or water bath until no odor of petroleum ether remains, cool to room temperature. Carefully remove any moisture or dirt from outside of flask and weigh. Repeat heating until constant weight is obtained.
7. Determine the moisture on ground sample as follows:
   a. Weigh 5 g. into a tared A.O.C.S. moisture dish.
   b. Slip the cover on bottom of the dish and place the dish in a forced draft oven. Dry at 130°C. ± 3°C. for 1 hour.
   c. Remove from oven and cover immediately. Cool in desiccator to room temperature and weigh.

$$\text{Moisture in ground sample, \%} = \frac{\text{Loss in weight} \times 100}{\text{Wt. of sample}}$$

E. Calculation

$$\text{Oil in ground sample, \%} = \frac{\text{Weight of oil} \times 100}{\text{Wt. of sample}}$$

The percentage of oil is calculated to any desired moisture basis with the following formula:

$$\text{Oil, desired moisture basis, \%} = \frac{F(100 - \text{\% moisture desired})}{100 - \text{\% moisture in ground sample}}$$

F = % oil determined in ground sample.

It is proposed that the present A.O.C.S. Official Methods Ba 1-6, inclusive, for the sampling and analysis of oilseed meals be revised to provide for the sampling and analysis of linseed meal. The changes considered necessary are given in the recommendations.

It is recommended:

1. That the proposed methods for the sampling of flaxseed, and the determination of moisture and volatile matter and of oil content of flaxseed, be adopted as tentative.
2. That the present A.O.C.S. Official Methods Ba 1-6, inclusive, be revised to provide for the sampling of linseed meal and the determination of moisture and volatile matter, oil content, protein, ash, and crude fiber in linseed meal be continued as official. The recommended revisions are:

a) Sampling Ba 1-38. Include the Boerner sampler, 36-pocket size, in addition to the Jones sampler, riffle type, 8 x 10 in., in the required apparatus. Specify the "bag trier" as one of sufficient length to reach from the top to the bottom of the bag.

b) Moisture and Volatile Matter Ba 2-38. Include the Boerner sampler, 36-pocket size, in the required apparatus. Specify that a 2-g. sample be dried at 130° ± 3°C. as for soybean oil meal.

c) Oil Content Ba 3-38. Require the use of a U. S. No. 30 sieve in preparing linseed meal for analysis, and include such a sieve in the list of apparatus required.

d) Protein Ba 4-38. Define protein as Nitrogen × 6.25 in the definition.

e) Include linseed meal in the "Scope" of the methods for the determination of moisture and volatile matter, oil content, protein, ash, and crude fiber.

J. C. KONEN, chairman
R. W. CORNELL
W. A. MOE
G. N. WALKER

Automatic Sampling of Bulk Oilseed Meals

THE sampling of bulk cars of meal is difficult, tedious, and expensive whether this is undertaken at the time of loading or upon delivery to the buyer. Disagreements have been frequent between buyer's and seller's samples, due to the tremendous difficulty of securing identical samples before and after transit.

The purpose of this study is to determine the feasibility of using automatic sampling devices for drawing official samples of oilseed meals at the point of origin for settlement purposes.

Two types of such equipment were available for this study at the Augusta, Georgia, and Corinth, Mississippi, mills of the Buckeye Cotton Oil Company. In both cases a traveling cup cuts the falling stream of meal at timed intervals. At the former mill the cup travels in a circular path while at the latter it travels in a straight line reversibly through the stream of the meal. In each instance the proportionate amount of sample can be varied by regulation of the interval timer, and provision is made mechanically for accumulating the portions drawn at each passage of the cups through the stream of the meal.

A single procedure was used at the two mills for testing the reliability of each installation on two types of meal, solvent-extracted at Augusta and hydraulic at Corinth. Three lots of 600 sacks each were sampled automatically at each mill, which accumulated 30 lbs. for each 600-sack lot in each instance.

In order to secure a reliable sample for comparison, the meal was sacked, and each fifth sack of each 600-sack lot was probed, using a Seedburo 78A probe, from one top corner to the diagonally opposite bottom corner. Approximately 25 pounds of meal were obtained by this method for each lot of sacked meal.

Each sample was then mixed in a large McClellan mixer. After mixing, the mixer was placed with the discharge down. A 1-gal. container was placed under the discharge, coupled to the discharge opening with a suitable baffle so that when the meal was dumped it passed directly into the 1-gal. container. After the container was filled, the mixer was closed. This procedure was followed in order to avoid possible segregation.

TABLE I

Analysis of Cottonseed Meals Sampled by Automatic and Probe Methods

Laboratory	A 1 A		A 1 P		A 2 A		A 2 P		A 3 A		A 3 P	
	H_2O	NH_3	H_2O	NH_3	H_2O	NH_3	H_2O	NH_3	H_2O	NH_3	H_2O	NH_3
	%	%	%	%	%	%	%	%	%	%	%	%
1	7.80	7.34	8.20	7.15	7.40	7.39	7.40	7.41	6.50	7.34	6.50	7.30
2	7.73	7.28	7.94	7.17	7.25	7.47	7.25	7.50	6.28	7.35	6.32	7.39
3	7.28	7.36	7.60	7.15	6.58	7.49	6.83	7.38	5.80	7.28	6.00	7.32
4	7.90	7.33	8.20	7.19	7.30	7.41	7.40	7.39	6.40	7.30	6.50	7.20
5	7.70	7.33	8.09	7.21	7.26	7.45	7.22	7.36	6.28	7.30	6.33	7.23
6	7.98	7.31	8.35	7.17	7.42	7.45	7.55	7.40	6.56	7.33	6.54	7.36
Average	7.73	7.33	8.06	7.17	7.20	7.44	7.28	7.41	6.30	7.32	6.37	7.32
Average (8% H_2O)		7.31		7.18		7.38		7.35		7.19		7.19

Laboratory	C 1 A		C 1 P		C 2 A		C 2 P		C 3 A		C 3 P	
	H_2O	NH_3	H_2O	NH_3	H_2O	NH_3	H_2O	NH_3	H_2O	NH_3	H_2O	NH_3
	%	%	%	%	%	%	%	%	%	%	%	%
1	7.30	8.25	7.40	8.23	7.40	8.22	7.40	8.15	7.00	8.30	6.90	8.28
2	6.58	8.23	6.82	8.29	6.76	8.16	6.67	8.20	6.27	8.27	6.26	8.37
3	6.25	8.28	6.70	8.22	6.45	8.22	6.30	8.22	6.20	8.27	5.95	8.21
4	6.90	8.23	7.10	8.23	7.00	8.20	7.10	8.23	6.50	8.28	6.66	8.26
5	6.56	8.25	6.73	8.22	6.69	8.25	6.63	8.21	6.22	8.33	6.19	8.35
6	6.67	8.20	6.86	8.22	6.75	8.28	6.68	8.23	6.29	8.41	6.21	8.30
Average	6.71	8.24	6.94	8.24	6.84	8.22	6.80	8.21	6.41	8.31	6.35	8.30
Average (8% H_2O)		8.13		8.15		8.12		8.10		8.17		8.15

Prefix A = Augusta. Prefix C = Corinth. Suffix A = Automatic. Suffix P = Probe.

The entire 1-gal. sample was then ground to approximately 20 mesh, mixed in a small McClellan mixer, and 6-oz. samples secured for distribution to the subcommittee members for analysis.

All samples were identified as to source and method of sampling. They were analyzed for moisture and nitrogen by the members of the subcommittee, using the A.O.C.S. Official Methods. The average results for the ammonia equivalent of the nitrogen content, calculated to moisture basis of 8%, show excellent agreement between the automatic and probe sampling (Table I).

It is demonstrated by the analytical data that the two types of automatic sampling tested provide samples of sufficient accuracy for use as official samples for sampling bulk oilseed meals at point of loading.

The subcommittee also analyzed a series of samples, comparing automatic and probe sampling of soybean meal at the Decatur, Illinois, plant of Spencer Kellogg and Sons. Automatic sampling in this instance is accomplished by installing a ¾-in. pipe in the scale hopper at the sack filling station of the plant, with the cap on the pipe drilled to provide a 3/16 in. hole to admit a portion of the meal as the hopper is filled. Samples were obtained from three lots of meals as being bagged automatically and by probing the bags after filling. The analytical results are given in Table II. While this method of automatic sampling may not be applicable to sampling bulk car loading, it could well be considered for obtaining official samples of meal at the time of bagging them.

Recommendations

1. That a procedure be included in A.O.C.S. Official Method Ba 1-38 for the automatic sampling of oilseed meals at the time of loading for bulk shipments at such time as the Uniform Methods Committee may order with the approval of the Society.
2. That the description of a method for automatic sampling a falling stream of meal should include:
   a) Basic specifications of an automatic sampler found satisfactory by test and experience with the several oilseed meals.
   b) Specification of quantity of sample to be taken per car.
   c) Specification of a satisfactory mixing device.
   d) A requirement that the reduced sample be ground to a specified fineness before final reduction to the sample size required by the laboratory.
   e) Provision for packaging the samples to avoid gain or loss in moisture.

G. C. HENRY, chairman
E. C. AINSLIE
M. W. DIPPOLD
T. J. POTTS
T. C. SMITH

Reports of the subcommittees have been reviewed by the Seed and Meal Analysis Committee and given unanimous approval:

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L. R. BROWN
F. R. EARLE
E. B. FREYER
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TABLE II

Analysis of Soybean Meals Sampled Antomatically and With a Probe

Laboratory	D 1 A		D 1 P		D 2 A		D 2 P		D 3 A		D 3 P	
	H_2O	NH_3	H_2O	NH_3	H_2O	NH_3	H_2O	NH_3	H_2O	NH_3	H_2O	NH_3
	%	%	%	%	%	%	%	%	%	%	%	%
1	9.60	9.01	10.90	8.78	11.40	8.95	11.40	8.79	11.10	8.95	11.00	8.92
2	9.19	9.08	10.86	8.84	10.84	8.97	10.80	8.86	10.57	8.98	10.53	9.00
3	8.20	9.12	9.60	8.80	10.00	9.00	9.95	8.78	9.75	9.07	9.75	8.97
4	9.00	9.13	10.50	8.85	10.60	8.97	10.70	8.92	10.40	9.01	10.60	8.94
5	9.08	9.03	10.63	8.87	10.77	9.00	10.85	8.79	10.47	9.03	10.67	8.87
6	9.58	9.09	11.10	8.83	11.10	8.98	11.20	8.86	10.80	9.06	10.90	8.92
Average	9.11	9.08	10.60	8.83	10.79	8.98	10.82	8.83	10.52	9.02	10.58	8.94
Average (12% H_2O)		8.79		8.69		8.86		8.71		8.87		8.78

Prefix D = Decatur. Suffix A = Automatic. Suffix P = Probe.