

METHOD

APPARATUS

Mechanical Lint Mixer

The action of the mixer is to throw hull pepper and dust back into the sample continuously during the picking and mixing operation. It consists of a drum made of copper with its cylindrical surface and 3 baffles running from end to end and attached to the inside surface of the cylinder. The drum is mounted on ball bearings on a brass shaft; the shaft mounted on ball bearings pillar blocks. Attached to the shaft are 24 radical brass spikes which revolve inside of the drum. The drum is turned at 6 R.P.M. clockwise and the spikes revolve at 150 R.P.M. counterclockwise. The capacity of this mixer is up to 5 pounds of lint.

Balances

- (1) A balance sensitive to .01 grams.
- (2) A balance sensitive to .001 grams.

Pots

Galvanized iron (glue) pots with a capacity of 1000 cc are used for digesting samples. Lids are made for the pots with one-eighth inch iron or nickel plate, with a three-quarter inch hole to one side of center. This hole is covered on the bottom with 60 mesh copper or nickel wire. An asbestos sheet one-sixteenth inch thick covers the entire bottom of the iron or nickel plate with the exception of the hole. Another plate (with hole matching the first) one-sixteenth thickness, is cut of a smaller diameter to fit inside the pot and holds the asbestos and screen wire in place by being bolted through the asbestos to the upper plate. The asbestos acts as a gasket around edges of the pot. An asbestos flap cover is made for the top of the hole that closes when steaming the digester and opens when the pressure is released. The top is fastened securely on the pot with a screw clamp.

Digester

A digester, or autoclave, with a working pressure of 105 pounds per square inch (341 degrees Fahrenheit) of steam.

Mechanical Washer

The principle of the washer is to suspend the cooked sample of lint or fibre in water, subject it to the beating action or tiny jets of water to separate the hulls from the fibres and finally drain off those fine partly cooked hulls with the cooking solution. This is accomplished by a slow end over-end rotation of a washing cylinder, 8½ inches long, 6½ inches in diameter, one end of which has a standard 60 mesh nickel screen, the other being closed except for a small one-half inch. 60 mesh, screen-covered opening which permits air to enter during draining. The cylinder turns on an axis through which a one-eighth inch brass water line enters. This brass lint has 44-1/32 inch openings, spaced regularly along the entire length. Water enters at a constant pressure of 22 pounds per square inch. During one complete rotation the cylinder becomes about one-half to three-quarters full of water, which drains out each time the screened end is turned downward. A deflector plate ¾ inch above the 60 mesh screen prevents water from spraying over the operator.

The cylinder during operation rotates at 4 R.P.M., being driven by an electric motor and reducing gear. A clutch arrangement permits disengagement of the cylinder and driving mechanism at any desired point.

Drying Ovens

Either a gravity or mechanical convection oven with a temperature range of 105 to 110°C. If a gravity convection oven is used it should give uniform temperature throughout the oven. Unless these ovens are well insulated wide ranges in temperature will be found. If a mechanical convection oven is used the air velocity across the sample should not be over 60 feet per second.

REAGENTS

Caustic Solution

A sodium hydroxide solution of exactly 1.0 per cent concentration using C. P. or U. S. P. caustic.

PROCEDURE

Laboratory Preparation of Sample

Either a gravity or mechanical convection oven the mechanical mixer. The latter gives better mixing, is quicker, gives less dust and gives possibly less loss of hull pepper. Either mixing procedure, however, can be used.

The mechanical lint mixing is carried out as follows: up to 5 pounds of lint or hull fibre is put in the mixer and the door closed. The mixer is rotated from 3 to 5 minutes depending on the length of the fibre. For second cut and hull fibre 3 minutes is sufficient. For mill run the machine should be run 5 minutes. After mixing two samples are taken out, one for moisture and one for yield determination.

In hand mixing the whole sample is pulled apart by hand and the lint mixed gently until uniform mix is obtained. Too much agitation causes loss of hull pepper and must be avoided. In mixing the hull pepper is worked back into the sample. Take part of the sample for moisture and part for yield determination. If the conditions in the laboratory are such that the sample will dry out during above division, a moisture test should be taken as soon as emptied, by taking small parts through-out the sample. This condition can best be established by checking moistures before and after mixing to determine correct procedure for each laboratory.

Moisture Determination

About 25 grams of the lint sample are put in a tared aluminum or tinned iron weighing can. These small cans must have air-tight tops, with a capacity of about 30 cubic inches. The sample is weighed on a balance that weighs to the third decimal place and the weight recorded. It is dried bone dry in a mechanical or gravity convection oven, as specified under apparatus, at 105 to 110 degrees Centigrade for 4 hours. If a mechanical convection oven is used, only a very slow current of air should be used. If a gravity convection oven is used care must be exercised that free circulation of air is allowed through samples and around thermostat so that a uniform temperature is maintained throughout

the oven. After drying, the sample should be cooled in a desiccator. After cooling, weigh and calculate the loss in weight as per cent moisture. Moistures should be run in duplicate unless the analyst is able to get identical checks on the composite sample.

Mixing Lint and Caustic Solution

Exactly thirty-five grams of the final sample are weighed on a balance that is accurate to the second decimal. It is transferred to the pot and 525 cc of the one per cent caustic solution is added, pressing the lint down as the addition of caustic is made, with a strong glass flask, bottle, or kitchen potato masher, to insure a good wetting-out. The mixture is then stirred with a glass rod to complete the mix, as a good mix is essential to this test. The lids are fastened on securely and the pots are put in the digester.

Digesting

Fasten the lid securely on the digester and bring the steam pressure up to 105 pounds pressure (341°F). The temperature should be watched closely. It is held at this temperature for three hours after the pressure is up and then the steam is blown off. Any lowering of the pressure during cooking will blow out some liquor thus giving worthless results.

Washing

A small amount of water is added to the cooked lint or fibre after it is removed from the autoclave in order to facilitate removal from the cooking pot. This mix is poured directly into the lower half of the washing cylinder. The cooking pot is given one rinse in order to assure complete transference of all fibres to the washer. The upper half of the washing cylinder is clamped on and the washer is started. The water is

turned on when the screened end reaches the bottom of its rotation. This eliminates plugging of the screen. The washing time begins when the water is turned on. The water is held constant at 22 pounds per square inch with a reducing valve. During the 5 minute washing period 3.9 to 4.0 gallons of water per minute should pass through the washing cylinder, which should be checked occasionally to see that the machine is working properly.

At the end of five minutes the water is cut off and the revolving motion stopped when the washing screen is near the bottom of its rotation. The cylinder should be slightly off vertical to assure rapid drainage. After the flow of drainage water has almost stopped, the water valve is again turned on for an instant to wash any adhering fibres from the walls of the cylinder. The lower half of the washer with its accompanying lint or fibre is taken off and the stock on the screen carefully removed and squeezed hard by hand. The screens in the end of the washers should be inspected at least once a month to insure that no plugging or sagging has taken place.

Drying

The wet sample is dried in the ovens mentioned above at 105 to 110 degrees Centigrade over night (14 hours). The weighing can be used in lint moisture determination may be used, but good results are obtained by putting the squeezed samples alone in the oven in a screened partitioned tray and weighing these samples hot immediately on taking out. The same balance as used for lint moisture is used. From the dry weight the unit yield is calculated, as bone dry yield from the lint or fibre as received. In other words, the bone dry cooked sample weight is divided by the lint weight used (no correction being made for lint moisture) times 100.

A Note on Muscadine Grape Seed Oil*

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ALTHOUGH grape seed has a very limited use in this country, it is used extensively elsewhere in the paint and soap industries, as well as for edible purposes. Oils from the *Vinifera* and *Labrusca* groups have been studied; however, a review of the literature did not reveal any reference to muscadine grape seed oil. The Georgia Experiment Station has done considerable horticultural and chemical investigations of the muscadine (*Muscadinia rotundifolia* (Michx.) Small) grape which is native to the southern part of the United States. Along with these studies a few oils from grape seeds of the leading muscadine varieties have been examined.

The muscadine seeds (2 to 6 per cent of the whole fruit) were by-products from fermentation. After they were thoroughly cleaned, the seeds were air-dried, ground, and extracted with Skellysolve F.

The data on the oils from six muscadine varieties are given in Table 1. It may be noted that the glycerides, calculated from the iodine numbers and thiocyanogen values, are only approximations as other unsaturated fatty acids, possibly a small amount of erucic acid, might be present.

Upon comparing with the properties of the *Vinifera* and *Labrusca* oils, data obtained by Jamieson (1), Rabak (2), and many other workers, muscadine grape

seed oils have many similar properties and can probably be used interchangeably for many commercial uses.

Literature Cited

- (1) Jamieson, G. S., and McKinney, R. S., *Oil and Soap*, 12, 241 (1935).
- (2) Rabak, F., *Ind. Eng. Chem.*, 13, 919 (1921).

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TABLE 1.—MUSCADINE GRAPE SEED OILS

	Varieties					
	Hunt	Scupper-nong	Brownie	Creek	Stuckey	Yuga
Oil in seed, air-dry, per cent	13.9	12.9	12.5	12.4	13.6	12.4
Iodine no. (Wijs).....	132.0	129.0				
Thiocyanogen value	77.8	76.2				
Calculated glycerides—						
Linoleic, per cent	62.5	60.9				
Oleic, per cent.....	27.4	27.2				
Saturated, per cent.....	9.1	11.0				
Unsaponifiable material, per cent....	1.04	0.87				
N ₂₅ ^o						
D	1.47060	1.47130				
Acid Value (mgs KOH/gm.)	12.9	8.2				