

droguariaretic acid and gallic acid, that have been reported. The carry-over of the antioxidant properties into baked goods was determined by storage tests on piecrust at 38° and 63°C. The results show that the higher gallates have good protective action in baked piecrust and are much superior to gallic acid and propyl gallate.

These higher esters of gallic acid are readily soluble in fats. This factor is of great importance in commercial stabilization of fats.

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Effect of Drying and Storing Tung Seeds On Quality of the Oil and Milling Characteristics of the Seeds*

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ONE of the major problems with which the tung oil industry is confronted is that of drying tung fruits or seeds sufficiently to be milled or stored. As production is increased this problem becomes even more important. The tung fruits contain 60% to 70% moisture when they fall from the trees, and they cannot be stored in bins in layers more than about a foot in depth until they have dried to 15% to 20% moisture, nor can the oil be pressed efficiently from tung seeds until the seeds have dried to about 9% moisture. Drying to even 20% moisture requires several weeks in the orchard. Without the use of driers it is not possible to start the milling operations until about two months after the fruits have matured. Also, in some years rainfall is so heavy that it is almost impossible to dry the fruits sufficiently in the orchard to store or to mill them.

Since there is a considerable investment in the processing plant, it is desirable to run the mill as long each season as feasible, and in many seasons the mills have operated into June. The fruits need to be off the ground by the first of April at the latest so that field operations can begin, and some form of storage would be necessary to accomplish this and enable the mills to operate as late as June. These facts point out the need of being able to dry and store tung fruits or seeds.

Obviously, it would be desirable to remove the hulls before drying or storage because of the smaller amount of heat required for the drying and the smaller storage capacity required for the hulled fruits. Even if there should be a market for the dried hulls it probably would not be feasible to dry the

whole fruits before hulling because of the loss of kernel fragments in hulling fruits that are too dry.

In practice, most of the hulling has been done in the tung mills by disc hullers, and this type of huller in addition to removing all of the hulls ordinarily removes about half of the shells and breaks some of the kernels, the amount of breakage varying with conditions of the fruits and setting of the discs. A huller has been developed by the Agricultural Engineering Division of the Bureau of Plant Industry, Soils and Agricultural Engineering which breaks a low percentage of shells (5). This huller can be built small enough to be used as a portable huller or as large as it needs to be. The storage experiments reported in this paper were carried out on seeds that had been hulled in one of these portable hullers, on the assumption that if hulled seeds are to be stored such storage would be limited to seeds with the least possible proportion of broken shells.

Drying and storage are so intimately connected that it is almost impossible to study the latter without considering the former. Also, drying and storage conditions may have considerable effect on the behavior of the seeds in the screw press. For these reasons experiments were designed in which seeds were dried under controlled conditions, stored for different periods, and then processed in a commercial screw press, thus permitting studies on all of these phases with the same material.

In general, it is well known that in handling other oil seeds such as cottonseed there is a certain moisture content above which development of acidity takes place rather rapidly at ordinary temperatures. The upper limit of moisture content for safe storage of these seeds is about 13% (3). These same principles would presumably apply to tung seeds. It is thought

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that the critical factor is not so much the per cent moisture in the whole seed as the per cent moisture in the non-oil fraction of the seed. Since the oil content of tung seeds is much higher than that of cottonseed or soybeans, it is probable that the critical moisture content of tung seeds based on the whole seed is lower than that of cottonseed. It is also known that if tung seeds are broken or crushed, exposing more of the surface of the kernel to the atmosphere, acidity develops much more rapidly (4).

I. Time Required for Drying Tung Seeds at Different Temperatures and Effect of Drying on Quality of Oil

Since in commercial practice tung seeds with 6-9% moisture seem to process best in screw presses and since it was thought that seeds dried much below this moisture content would reabsorb moisture from the atmosphere, it was decided to dry the seeds only to 8-10% moisture before putting them in storage.

The tung seeds used in these storage experiments were hulled in a portable huller (5) which broke comparatively few of the shells. Batches were dried and stored in December and January. About 12% (by weight) of the seeds stored in December, but only about 7% of those stored in January, had the shells broken. Batches of these seeds large enough for milling tests (600 lbs.) were dried to a moisture content of approximately 10% in a pilot plant tray drier at the Southern Regional Research Laboratory in New Orleans, Louisiana. The drier was thermostatically controlled and was first brought up to the required temperature before loading. It had 20 trays which, filled to a depth of 2 inches, held about 600 pounds of seeds. The progress of drying was followed by determining the initial weight and moisture content of seeds on two trays and weighing these trays at intervals until their weight had dropped to that required for 10% moisture. Batches were to be dried at 212° and 158° F., but loading the drier at the higher temperature with cold seeds lowered the temperature to about 155° F., from which it rose only slowly as drying took place. The dried batches were stored in bags for different periods in a well ventilated shed. At the same time similar batches of seeds which had not been artificially dried were stored in bags in the same shed, and in bulk in a pen built on the floor of hardware cloth in the form of a 3-foot cube.

The first batches were dried and put in storage on December 3, when the seeds contained 17% moisture due to the unusually dry fall while the second batches were put in storage on January 30, when the seeds contained 26% moisture because of the recent heavy winter rains.

With 20-lb. to 40-lb. batches of seeds the drier could be kept at constant temperature throughout the drying period. In Table 1 are shown the rates of drying on a number of small batches at different temperatures, determined in March, 1945. The data on the small batches give a better idea of the rate of drying than do those on larger batches because the temperatures were constant on the former but not on the latter. Apparently, drying directly with the air from the gas burners is slower for a given temperature, probably because of the higher humidity of the drying air resulting from the moisture from combustion. This material had about half the shells broken

or removed. Unpublished data on hand show that seeds with shells removed or broken dry somewhat faster than do seeds with the shells unbroken. The drying time required in commercial driers may be considerable longer due to the influence of the liberated moisture on the drying rate.

TABLE 1.
Time Required to Dry Tung Seeds at Different Temperatures.¹

Temp. of drying	Method of heating air	Time to dry to 10% moisture	Time to dry to 6% moisture
° F.		min.	min.
175	Steam coils	80	150
190	Steam coils	50	90
200	Directly by gas	60	110
230	Directly by gas	30	50
260	Directly by gas	to 2.4% moisture in 60 min.
280	Directly by gas	to 1.7% in 75 min.

¹ Seeds were hulled in a disc huller, with most of the shells and many of the kernels broken. Air was forced vertically through trays of seeds. Original moisture content was 28%.

These data bring out the fact that because tung seeds are larger than ordinary grains or beans, a longer time is required for drying, from 1 to 2 hours being required to dry seeds from a moisture content of about 28% to 10% at temperatures of 175° to 200° F. Any continuous drier would have to be designed to allow for this long a period of drying.

TABLE 2.
Effect of Different Temperatures Used for Drying Seeds on the Quality of the Oil¹

Temp. of drying	Drying time	Final moist.	Brown heat test	Ref. Ind. at 25°C.	Acid Number	Color
° F.	min.	%	min.			
(Air temp.)	11.00	1.5188	good
175	165	5.6	10.75	1.5184	.3	good
190	330	.8	10.25	1.5180	.2	good
200	180	3.0	9.50	1.5180	.1	good
230	60	4.5	8.50	1.5181	.2	dark
260	60	2.4	9.25	1.5182	.2	dark
280	75	1.7	9.50	1.5182	.2	dark

¹ Samples of oil were pressed out of seeds in a small hydraulic press soon after they were dried. Original moisture content was 28%.

In Table 2 are shown the properties of the oil pressed from samples dried at different temperatures. These are the same samples used for obtaining the data in Table 1. Heating seeds as high as 200° F. has no detrimental effect on the quality of the oil in seeds. Heating to 230° F. and above did have a detrimental effect in that it produced a dark color in the oil. Polymerization of the oil was probably started as shown by the decrease in the time of the Brown heat test.

It was observed in drying wet seeds (26% moisture) at 212° F. that many of the shells were cracked by the drying. This would be objectionable in storing, as the cracked seeds would have a tendency to deteriorate faster than whole ones.

II. Effect of Storage on Quality of Oil and Milling Characteristics of Tung Seeds

Development of acidity, decrease in oil content, and behavior of the seeds in the screw press are the three factors that have to be watched in stored seeds.

At two-month intervals milling tests were made on the stored batches with a factory-size screw press by the procedure described by Holmes and Pack (2). In carrying out these tests, precautions were taken to see that the press used was operating smoothly on the regular factory material and the press was then

changed over to the test material with no interruption in operation. Samples of the meal, screw press cake, and oil were composited during the test and analyzed, and from these analyses and the weights of oil and cake the oil recovery was calculated. At the time milling tests were made on the stored material tests were also made on the regular material from the disc huller being currently processed in the mill in which these tests were made to obtain a comparison of the stored seeds with those handled according to current commercial practices. The results are summarized in Tables 3 to 6. In Table 7, are shown the oil contents of cleaned kernels from the stored batches on certain dates.

TABLE 3.
Moisture Content of Stored Tung Seeds.¹
A. Seeds stored Dec. 3, 1945.

	Date						
	Dec. 3	Dec. 13	Jan. 6	Feb. 5-7	Feb. 18	Mar. 14	Mar. 27-Apr. 2
	%	%	%	%	%	%	%
Dried at 155-190°F.....	10.6	8.7	7.7	7.4	7.4	8.6	7.9
Dried at 158°F.....	9.6	8.3	7.8	8.4	8.0	8.6
Dried at 150°F.....	9.3	9.1	7.0	8.8
Dried at 115°F.....	10.4	8.9	7.4	8.0
No artif. drying.....	17.0	12.7	8.6	9.0	7.8	9.0
No artif. drying, bulk storage.....	17.0	15.6	9.8	9.0	9.0

B. Seeds stored January 30, 1946.

	Jan. 30	Feb. 5-7	Feb. 18	Mar. 14	Mar. 27-Apr. 2
		%	%	%	%
Dried at 155-212°F.....	11.2	9.2	8.4	7.4	8.4
Dried at 158°F.....	12.1	10.0	7.8	8.7
No. artif. drying.....	26.0	19.4	14.8	10.8

¹ Stored in bags except where noted otherwise.

Samples of the various batches of stored material were also taken for moisture determinations between the dates of the milling tests. In Table 3 are shown the moisture contents of the stored material on different dates as compiled from the milling tests and from the extra samples taken for the purpose. In general, the moisture content of all the seeds which had been dried remained between 7% and 10% during the two to four months they were stored. The air-dried seeds also dried to about 10% moisture after two months in storage. After February there were slight increases in moisture content which presumably were due to blowing rain.

TABLE 5.
Screw Press Tests on Tung Seeds.¹

Date of test	Dried at 155-212°F. 11.2% moist. when stored. Bag storage.		Dried at 158°F. 12.1% moist. when stored. Bag storage.	No artificial drying. 26% moist. when stored. Bag storage.
	Feb. 6	Apr. 1	Apr. 1	Apr. 1
Meal				
(1) Wt., lbs.....	341.9	452.0	372.4	336.0
(2) % shells plus debris.....	33.2
(3) Ratio wt. shells to wt. kernels, %.....	58.0
(4) % moisture.....	9.2	8.4	8.7	10.8
(5) % oil, dry basis.....	40.9	41.0	41.3	42.6
Screw Press Cake				
(6) Wt., lbs.....	201.4	278.5	218.4	196.6
(7) % moisture.....	6.8	6.8	6.4	7.3
(8) % oil.....	5.6	8.8	4.5	7.8
(9) Wt., oil, lbs.....	11.3	24.5	9.8	15.3
Crude Oil				
(10) Wt., lbs.....	122.6	154.4	135.6	117.5
(11) % solids.....	5.6	5.9	3.7	4.3
(12) Wt. pure oil, lbs.....	115.7	145.3	130.6	112.4
Filtered Oil				
(13) Ref. index at 25°C.....	1.5180	1.5178	1.5179	1.5179
(14) Acid value.....	.6	1.3	1.0	4.1
(15) Oil recovery, % (12) / ((12)+(9)) × 100.....	91.1	85.6	93.0	88.0

¹ Each test is calculated for 1 hour's operation of press. Intact seeds put in storage January 30, 1946.

The oil content of the dry seed meal is shown in line 5 of Tables 4 to 6 as calculated from the weights and analyses of oil and screw press cake obtained during an hour's run. These data do not show a significant change in oil content. This is confirmed by analyses of cleaned kernels shown in Table 7, except that there is possibly a drop in the oil content of seeds dried at 155-212°F. and stored in January.

An examination of line 14 in Tables 4-6 shows that by April the acidity of the oil in all stored batches of seeds had increased slightly. That of the seeds stored in January without artificial drying was 4.1, but when seeds with broken shells and kernels were picked out and analyzed separately the acid value of the oil was found to be 26.0. An acid value of 4.1 is not too serious when it is remembered that the A.S.T.M. specifications (1) allow an acid value of 8.0 for raw tung oil although an acid value of 3.0 is specified if an oil of low acid value is desired. The undried seeds put into bag storage on January 30 were heavily coated with mold at the end of one month and also when they were run through the screw press at

TABLE 4.
Screw Press Tests on Tung Seeds.¹

Date of test	Dried at 155-190°F. 10.6% moist. when stored. Bag storage.			Dried at 158°F. 9.6% moist. when stored. Bag storage.		No artificial drying. 17% moist. when stored		
	Dec. 13	Feb. 6	Apr. 1	Feb. 6	Apr. 1	Bag Storage		Bulk Storage
						Feb. 6	Apr. 1	Apr. 1
Meal								
(1) Wt., lbs.....	321.0	368.4	383.7	367.5	350.4	329.4	352.4	359.3
(2) % shells plus debris.....	29.6	29.4	29.4	29.9	29.9	29.4
(3) Ratio wt. shells to wt. kernels %.....	48.0	47.0	47.0	48.0	46.0	47.0
(4) % moisture.....	8.7	7.6	7.9	7.8	8.6	8.6	9.0	9.0
(5) % oil, dry basis.....	46.7	45.6	44.2	45.0	43.8	43.7	43.0	44.1
Screw Press Cake								
(6) Wt., lbs.....	166.8	199.1	210.3	201.5	192.8	182.2	203.4	200.0
(7) % moisture.....	5.4	6.3	5.3	6.3	6.2	6.1	7.0	6.6
(8) % oil.....	4.5	4.4	4.2	4.4	3.6	4.4	5.0	4.8
(9) Wt. oil, lbs.....	7.5	8.8	8.8	8.8	6.9	8.1	10.2	9.6
Crude Oil								
(10) Wt., lbs.....	135.3	153.8	154.2	149.9	139.5	129.9	131.5	140.2
(11) % solids.....	4.3	4.7	4.5	4.3	4.4	5.0	2.9	3.9
(12) Wt. pure oil, lbs.....	129.5	146.5	147.3	143.5	133.4	123.4	127.7	134.7
Filtered Oil								
(13) Ref. index at 25°C.....	1.5186	1.5181	1.5181	1.5181	1.5181	1.5182	1.5181	1.5182
(14) Acid value.....	.5	.6	.8	.6	1.4	1.7	1.5	2.0
(15) Oil recovery, % (12) / ((12)+(9)) × 100.....	94.5	94.3	94.4	94.2	95.1	93.8	92.6	93.3

¹ Each test is calculated for 1 hour's operation of press. Intact seeds put in storage Dec. 3, 1945.

the end of two months. Most of these seeds had been wet in storage to some extent by blowing rain. The indications are that there would be no trouble from development of acidity in dried whole seeds kept in storage for as much as four months during the cooler part of the year.

TABLE 6.
Screw Press Tests on Tung Seeds.¹

Date of test	Dec. 13	Feb. 6	Apr. 1
Meal			
(1) Wt., lbs.....	297.9	309.7	287.7
(2) % shells plus debris.....	19.9	16.8	28.0
(3) Ratio wt. shells to wt. kernels.....	29.0	23.0	45.0
(4) % moisture.....	10.5	8.6	9.7
(5) % oil, dry basis.....	53.6	53.0	47.6
Screw Press Cake			
(6) Wt., lbs.....	139.7	140.4	148.6
(7) %moisture.....	8.1	6.8	7.1
(8) % oil.....	7.2	5.5	5.5
(9) Wt., oil, lbs.....	10.1	7.8	8.2
Crude Oil			
(10) Wt., lbs.....	138.3	152.3	121.8
(11) % solids.....	3.8	6.6	5.1
(12) Wt. pure oil, lbs.....	133.0	142.2	115.6
Filtered Oil			
(13) Ref. index at 25° C.....	1.5184	1.5182	1.5179
(14) Acid value.....	.9	1.0	2.3
(15) Oil recovery, %			
(12)			
$\frac{(12)}{(9)} \times 100$	92.9	94.9	93.4

¹ Each test is calculated for 1 hour's operation of press. Tests on regular material from the disc huller being processed currently.

The refractive indices of the oil (line 13 of Tables 4-6) from none of the batches changed significantly in storage and all values were well within the limits specified for tung oil by A.S.T.M. (1).

TABLE 7.
Effect of Drying Treatment and Storage on Oil Content of Tung Kernels.¹

Date of storage	Heat treatment	Storage	Date of analysis		
			Jan. 9	Feb. 26	Mar. 26
Dec. 3	none	bag	%	%	%
Dec. 3	none	bulk	66.8	67.3	67.1
Dec. 3	155-190° F.	bag	67.8	67.3	66.8
Dec. 3	158° F.	bag	68.2	68.2	67.6
Dec. 3	158° F.	bag	68.2	68.0	67.7
Dec. 3	150° F.	bag	68.1	67.9	67.9
Dec. 3	115° F.	bag	68.1	67.7	68.3
Jan. 30	none	bag	66.9	68.3
Jan. 30	155-212° F.	bag	66.3	63.9
Jan. 30	158° F.	bag	66.7	66.6

¹ Per cent oil in kernel is given on a dry basis.

In comparison with the regular material from the disc huller the seeds with few shells broken, stored in December, processed very efficiently as late as April, and the oil from them was still of very high quality. The seeds put in storage January 30, originally containing 26% moisture, processed less satisfactorily. The batches dried at 155-212° F. processed fairly well immediately after drying and the batches dried at 158° F. processed satisfactorily two months after drying. After two months the seeds dried at the higher temperature and those stored without artificial

drying processed very inefficiently. The acid value of the oil from the latter was also higher than desirable. It might be possible to manipulate a screw press so that seeds of high original moisture content, dried at 212° F. and stored, could be efficiently processed but storing such material should be done with caution until further information is available on the problem. The moisture content of the seeds put in storage in January without artificial drying was apparently too high for safe storage.

Summary and Conclusions

1. Tung seeds, hulled but with most of the shells intact, were artificially dried at two temperatures in December and January and put in storage in bags in a well ventilated shed, along with similar seeds which had not been artificially dried. At bimonthly intervals tests on stored seeds were made on a commercial screw press.

2. The acid value of oil in the intact seeds put in storage at 10% moisture content early in December and late in January had risen only slightly, from .5 to 0.8-1.5, by the following April.

3. Intact seeds dried to about 10% moisture at temperatures of 158° F. and about 172° F. (155-190° F.) processed in the screw press as well the following April as they did when put in storage in early December, and there was no deterioration in the quality of the oil.

4. Intact seeds which were dried from 26% moisture to about 10% moisture in late January at 155-212° F. (the temperature remained at 212° F. for at least an hour) did not process efficiently after two months' storage, nor did the material stored at the same time without artificial drying.

5. Caution should be used in storing seeds for long periods after drying at temperatures approaching 212° F. or with high moisture contents (20% or above) until more experience is available.

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