A Preliminary Study of the Effect of Moisture Content, Rolling, and Cooking of Cottonseed Meats on the Chemical Properties of Hydraulic-Pressed Meals

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IN conventional methods of processing cottonseed for oil and meal, cooking to some degree is essential for the efficient removal of the oil. What takes place in the meats during cooking has a direct influence on the quality of the final cottonseed products. The color and refining loss of the oil and the content of free gossypol (10) and the protein value of the meal are affected by the conditions of cooking.

Chemical studies have indicated that free gossypol is the material in cottonseed meal which limits the amount that may be used in diets of swine and poultry. In order to broaden the utilization of cottonseed meal in livestock feeding it is necessary to reduce the content of free gossypol in the meal to a level where it will not interfere with the growth of these animals. Present indications are that when the free gossypol content is reduced to values close to .03%, this may be sufficient to allow the meal to be used in unrestricted quantities in diets for nonruminants.

Cooking at high temperature for a long time reduces the free gossypol in the meal to values close to .03%. The disadvantage of accomplishing the reduction in this manner is that excessive heat alters the chemical quality of the protein (7) and would probably lower the nutritive value of the meal (5). Therefore the development of a method of processing cottonseed which would result in a low enough free gossypol content without requiring severe cooking conditions would have some advantages.

The approach reported in this paper was to consider the variables of cooking which influence the reduction of gossypol and to determine whether the changes could be brought about by operations other than cooking, at least to an extent to permit milder temperatures during cooking.

It is known that cooking under the proper conditions ruptures the walls of glands in which the pigments of cottonseed are contained; and that the pigments liberated from the glands combine with the protein or other constituents of the meats. "Free gossypol" (readily extractable gossypol) thus becomes converted to bound gossypol (1, 8, 11). One approach to the problem would be to use means of rupturing the pigment glands other than in the cooking step so that milder temperatures could be used in cooking.

In work on the control of cooking variables in mechanical methods of processing cottonseed (4, 6), meals were produced whose content of free gossypol was very low (0.01-0.04%). Moreover only moderate chemical alteration occurred in the protein. Physical and chemical analyses of the cooked meats as compared to those of the pressed meals (6) indicated that the larger part of the reduction in free gossypol had taken place in the screw press. Laboratory experiments showed that the hydraulic press was ineffective in rupturing the glands (4). This fact suggested that the shearing action in the screw press was largely responsible for the gland breakage. It was desired therefore to devise means for breaking glands in meats for hydraulic pressing and solvent extraction which would have the same effect of gland breakage as the shearing action of the screw press and permit the reduction of gossypol to take place under mild conditions of cooking.

In the present investigation a method of rolling cottonseed flakes which results in breakage in the flaked meats of more than 50% of the glands was devised. It is believed that in only a few hydraulic mills is this degree of gland breakage approached and that conventional flaking ordinarily breaks only a small percentage of glands (3). It was expected that the prebreakage of glands would permit milder cooking to rupture the remaining glands and to reduce free gossypol by binding with other constituents.

In a second part of this work data were obtained on the physical and chemical properties of the meals produced by hydraulic pressing from meats prepared with and without the pre-breakage of the large amount of glands. Hydraulic-pressed meals produced from meats of identical moisture content under identical conditions of cooking and pressing distinctly differed in contents of free gossypol without any significant effect on other physical and chemical properties, according to whether they had been produced from flakes which had or had not been specially rolled to break glands.

Experimental

Materials and Preparation. Delinted cottonseed (Thorndale, Tex., 1950 crop), which had been stored for six months, was hulled with a bar huller. The whole meats and fines were combined and adjusted with hulls so that the resultant mixture yielded 41% protein in the press cake. The meats were freshly prepared before each experiment and had an original moisture content of approximately 6.6%.

To prepare meats for the rolling experiments, they were preflaked on smooth rolls set at 0.010-in. clearance to create a larger area for moistening, and were then divided into four lots and moistened with water to four levels, respectively, 7.4, 10.8, 14.0, and 19.8%.

The desired moisture level was attained by controlling the amount of water added by use of a calibrated spray from a Monarch [3] No. 6.00 nozzle, which delivers 3.68 g.p.h. at a line pressure of 50 p.s.i.g. During moistening the meats were agitated by use of a rotary

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[3] The mention of trade products does not imply that they are endorsed by the Department of Agriculture over similar products not mentioned.

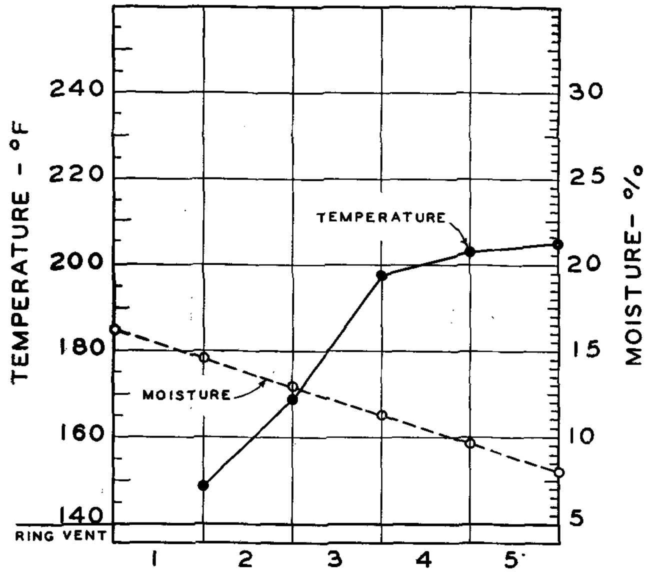

FIG. 1. Variations in moisture contents and temperatures of flakes during cooking for Runs 1 and 2. Jacket pressures 8 P.S.I., stack blower on.

pulp mixer of 33 cu. ft. capacity and a shaft speed of 33 r.p.m.

Rolling Conditions. Immediately after the flakes were moistened, portions of the four lots were rolled by means of conventional smooth flaking rolls, corrugated cracking rolls, and 5-high rolls. The first two types were tried with different clearances: .001, .004, and .010 in. spacing.

The smooth flaking rolls were 12 inches long by 12 inches in diameter and had identical speeds, 220 r.p.m. They were spring-loaded at 10,000 lb. Stationary metal blade scrapers were used to clean them of adhering material.

The corrugated rolls, also 12 in. long by 12 in. in diameter, had differential speeds of 1:2.35, the faster roll moving at 220 r.p.m. The rolls were spring-loaded at 10,000 lb. Stationary brushes were used in cleaning them.

The top two of the 5-high rolls were corrugated, and the bottom three were smooth. All were 12 inches long, the top four being 14 inches in diameter, and the bottom roll, 16 inches. Roll speed was 178 r.p.m. Weighted blade scrapers were used in cleaning.

In the preparation of meats for cooking and hydraulic pressing the severe (5-high) and the mild (smooth, 0.005 in. clearance) rolls were used.

Determination of Degree of Gland Breakage. The rolled meats were analyzed for gland breakage by subjecting them to extraction with hexane. With hexane extraction the gossypol which has been released from the ruptured glands is either largely removed in the miscella or is bound on the meal. The extracted, dried meal was analyzed for residual free gossypol, using the method of Pons and Guthrie (10) and for total gossypol using that of Pons *et al.* (11). The degree of gland breakage for a particular sample was calculated by dividing the free gossypol content of the meal after extraction by the free gossypol content of the control meal (prepared from unrolled meats) after extraction, substracting the quotient from 1.00, and multiplying the difference by 100.

The meats were extracted as follows. To a 240-g. sample of meats in a 3-necked flask of 3-liter capacity, 460 cc. of hexane was added. These proportions are similar to those used in a commercial extractor; on a weight basis the average oil-hexane ratio would be 1:4, assuming 33% lipides in the meats. The flask was immersed in a constant temperature bath maintained at 135°F. and the mixture was mechanically stirred at 100 r.p.m. for 20 minutes. Then the slurry was poured onto a Büchner funnel and filtered, under vacuum. The residue was washed twice, first with 300 cc. of fresh oil-hexane (1:4) miscella and then with 200 cc. of pure hexane. The meal was air-dried and analyzed.

The gland breakage determined by the extraction test was compared to that which had been observed in unextracted samples under a 54-power wide field microscope, a gland having been considered unbroken when there was no visible diffusion of pigment into the surrounding meal tissue or visible rupture of the wall. In making the determinations of broken glands the estimated percentages on each of 10 mounts had been averaged. The values obtained by the microscopic and the extraction tests were similar, but those found by the latter method are used in the discussion below since they depend on chemical analysis rather than on the analyst's estimation.

Cooking and Hydraulic Pressing. Four pairs of pilot-plant cooking and hydraulic pressing experiments were carried out on two portions of freshly prepared meats which had been preflaked, moistened to 14%, and rolled, respectively, under severe and mild conditions. Before cooking, the degree of gland breakage of the two portions was determined.

The meats of both runs of each pair were cooked by identical procedures so that any differences noted in the analyses of the press cake could be attributed to the differences in the rolls and rolling conditions. The

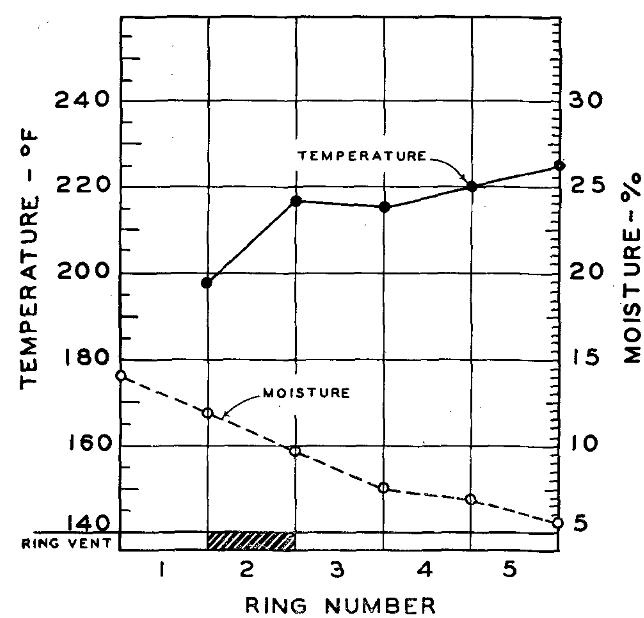

FIG. 2. Variations in moisture contents and temperatures of flakes during cooking for Runs 3 and 4. Jacket pressures 25 P.S.I., stack blower on.

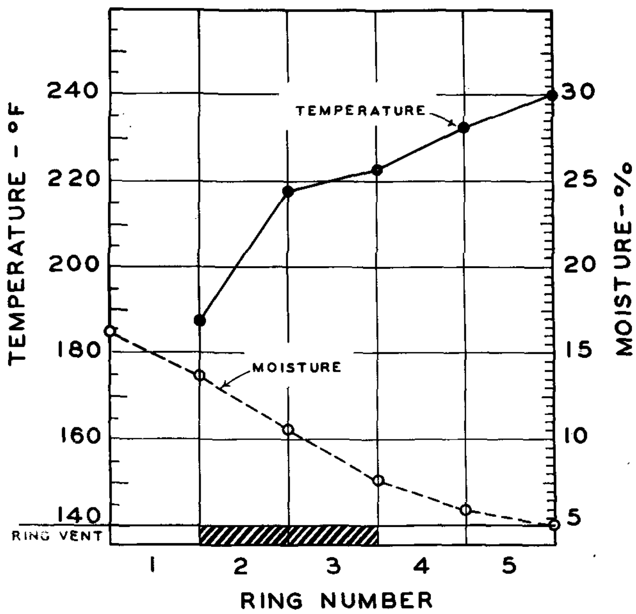

FIG. 3. Variations in moisture contents and temperatures of flakes during cooking for Runs 5 and 6. Jacket pressures 35 P.S.I., stack blower off.

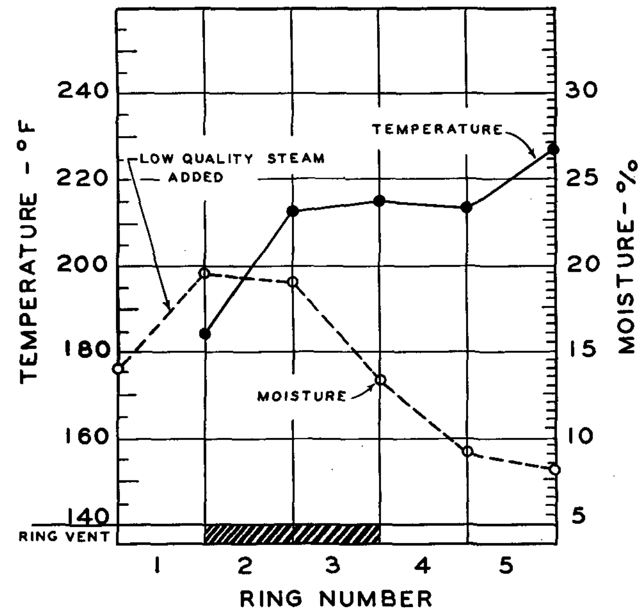

FIG. 4. Variations in moisture contents and temperatures of flakes during cooking for Runs 7 and 8. Jacket pressures 25 P.S.I., stack blower on.

cooking variables in the four runs were the maximum temperature of cooking (highest in any run 240°F.), the amount of moisture added during the cooking operation, and the rate of moisture lost during cooking. The cooking conditions are shown in Figures 1-4.

In each experiment the charge was cooked for one hour, while being agitated continuously. A vertical-type French [3] cooker was used. It consisted of 5 rings, each 22 in. in diameter and 18 in. high. Each ring was separately jacketed for indirect heating with steam, and was equipped with separate steam pressure regulating valves and gauges, traps, and recording thermometers which indicated the temperature cycle of the meal in the chamber; also with a steam valve which permitted direct addition of steam to the space above the meal in the cooking chamber. The stirrers in each chamber had a common shaft, rotating at 38 r.p.m. The contents of a ring were discharged into the ring below it through a small opening in the bottom which was operated manually, a few minutes elapsing between the initial and final discharge. In addition, rings 2, 3, and 4 had a vent opening, to which a blower was attached to provide the option of operating the cooker under natural or forced draft.

A French [3] cake former which was situated at the discharge of the cooker permitted cakes 8 x 18 x 2¼ in. to be formed in press cloths. Nylon cloths were used. A French [3] hydraulic press was used which contained six press box sections, and whose ram was 9 inches in diameter and could be operated at a hydraulic pressure up to 5,000 lb. per square inch.

Pressing operations required 30 minutes. Pressure was brought up slowly to 800-1,000 pounds per square inch ram pressure in the first five minutes. Caution was taken to avoid increasing the pressure too quickly. The pressure was then increased to 2,500 pounds per square inch ram pressure and held constant for 10 minutes; then was increased to the maximum, 4,600 lb. and held there for the remaining 15 minutes. The pressure was then released and the cake discharged. The cake from each press box was about 8 in. wide and 18 in. long, ¾ in. thick, and weighed about 5 lb.

Samples of the cooked meats and press cake were analyzed for moisture by drying for two hours at

TABLE I
Effect of Moisture Content and Rolling on Gland Breakage

Rolling Conditions	Free Gossypol Content of Extracted Meal [1] %				Total Gossypol Content of Extracted Meal %				Degree of Gland Breakage by Extraction Test %				Degree of Gland Breakage by Microscopic Test %			
Moisture content—%	7.4	10.8	14.0	19.8	7.4	10.8	14.0	19.8	7.4	10.8	14.0	19.8	7.4	10.8	14.0	19.8
No rolling-control	0.80	0.80	0.80	0.59	0.81	0.76	0.90	0.72	0	0	0	0	5	5	5	10
Smooth rolls clearance, in.:																
.001	0.63	0.82	0.63	0.40	0.78	0.85	0.79	0.57	21	0	21	32	5	5	10	50
.004	0.76	0.86	0.74	0.44	0.84	0.79	0.87	0.66	5	0	7	25	5	5	5	35
.010	0.81	0.79	0.80	0.54	0.93	0.86	0.90	0.72	0	1	0	9	5	5	5	20
Corrugated rolls clearance, in.:																
.001	0.61	0.61	0.44	0.29	0.75	0.79	0.73	0.67	24	24	45	51	15	20	35	70
.004	0.68	0.70	0.64	....[2]	0.76	0.87	0.87	....[2]	15	13	20	....[2]	5	15	20	....[2]
.010	0.72	0.77	0.70	....[2]	0.74	0.88	0.86	....[2]	10	4	13	....[2]	5	5	15	....[2]
5-High rolls	0.56	0.36	0.37	....[2]	0.73	0.69	0.71	....[2]	30	55	54	....[2]	20	30	40	....[2]

[1] Free-gossypol (gossypol or gossypol-like pigments readily extractable with aqueous acetone) in meal following extraction of the rolled material by hexane.

[2] No data; material was too moist to go through rolls.

101°C. in a forced-draft oven. They were analyzed for free gossypol (10), total gossypol (11), and nitrogen solubility (9). The press cake was analyzed also for lipide content (2).

Results

Effect of Moisture and Rolling on Gland Breakage. The data in Table I show the effects of the three types of rolls on the flakes moistened to the different levels. The degree of gland breakage increased with increasing moisture content of the flakes, regardless of the type of rolls used. Not more than 54% gland breakage was accomplished under any of the conditions.

The increase in gland breakage did not seem to follow regularly the increase in moisture content. For each type of roll the degree of breakage seemed to fall into two levels throughout the moisture range. There seemed to be two moisture ranges, one at which the degree of gland breakage was at a constant and lower level, and another at which it was at a constant and higher level. Corrugated rolls set at a spacing of .001 in. effected approximately 25% breakage at 7.4 and 10.8% moisture and 45 to 54% breakage at 14.0 and 19.8% moisture.

The 5-high rolls accomplished the greater degree of gland breakage at much lower moisture content, 10.8%, than either the smooth or the corrugated rolls at 19.8% and 14.0%, respectively.

The results (not shown in the table) with flakes moistened to 14% for the cooking and pressing of two portions which had been rolled under contrasting conditions showed that those rolled under the severe conditions (5-high, closely spaced) suffered 54% gland breakage, as compared to 5% for the mild rolling conditions.

Chemical Properties of Cooked Meats and Press Cakes. The analyses of the cooked meats and press cakes are given in Table II. It is shown that in every pair of experiments the more severe rolling operations resulted in much smaller remaining amounts of free gossypol in cooked meats or press cakes. The press cake produced in Experiment 8 had a free gossypol content of .03%, which compares favorably to meals produced by other processes and which have been fed in unrestricted quantities to swine and poultry (5).

In view of the possibility that reduction of free gossypol can take place by reaction of the gossypol released from pigment glands with the meal constituents, the advantages shown to be the result of more severe rolling could be attributed completely to more efficient rupture of pigment glands prior to cooking. More severe rolling however also resulted in greater tissue disintegration, which allowed for better mixture of all constituents and possibly a greater reaction, and it is quite possible that this fact in part accounts for the advantage gained by the more drastic rolling.

The differences noted in Table II in the free gossypol content of cooked meats and press cake in each pair of experiments cannot be attributed to differences in cooking conditions which may have inadvertently arisen in each experiment since with little exception the moisture content of each pair of cooked meats was the same and the reduction in nitrogen solubility, which is some measure of the heat damage to the protein, was equal. These facts seem to indicate that any differences in the chemical properties of the cooked meats or press cake would have to be attributed to the differences in the rolling conditions.

A difference in lipide content between the press cakes in the mildly and severely rolled meats in Experiments Nos. 5 and 7 and 6 and 8, respectively, even though the pressing conditions in each case were similar, is noted. It is not possible however on the basis of only two pairs of experiments, to attach any significance to the relation indicated between rolling procedure and lipide content in the press cake. Although no work is reported on the properties of the oil, it is recognized that consideration of the oil is imperative in determining the effect of rolling on processing conditions, and work on this phase is in progress.

Summary and Conclusions. The effectiveness of closely set five-high rolls, corrugated rolls, and smooth rolls for accomplishing gland breakage in cottonseed meats has been compared. Severe (5-high rolls) rather than mild (smooth rolls) conditions resulted in the large amount of gland breakage. A maximum gland breakage of 54% was generally obtained at moisture contents of 14% or above in the meats.

The degree of gland breakage was determined by an empirical method using hexane extraction. Samples having a larger amount of gland breakage had a much lower content of free gossypol after cooking and pressing.

Under the best conditions of rolling and cooking, in which the temperature of cooking did not exceed 225° F., a hydraulic-press meal was obtained with a free-gossypol content of .03% and with 40% nitrogen solubility.

These results indicate that it may be possible to carry out the processing necessary to produce meals of low free-gossypol content prior to the steps that are

TABLE II

Some Chemical Properties of Cooked Meats[a] and Press Cake from Pilot-Plant Hydraulic Process

Experiment No.	Rolling Conditions	Free Gossypol[b]		Total Gossypol[c]	Lipides	Nitrogen Solubility[d]		Moisture[e]	
		Cooked Meats	Press Cake	Press Cake	Press Cake	Cooked Meats	Press Cake	Cooked Meats	Press Cake
		%	%	%	%	%	%	%	%
1	Mild	0.33	0.30	.86	9.2	62	61	7.1	9.1
2	Severe	0.17	0.19	.87	9.4	59	58	7.2	9.2
3	Mild	0.17	0.18	.93	7.4	54	53	6.0	7.6
4	Severe	0.07	0.06	.94	5.3	53	53	4.9	6.3
5	Mild	0.16	0.17	.98	7.3	49	47	5.0	6.3
6	Severe	0.07	0.08	.96	5.5	45	48	4.9	6.4
7	Mild	0.09	0.08	.86	7.3	42	43	7.5	9.1
8	Severe	0.05	0.03	.82	5.4	46	41	7.8	10.4

[a] Cooking conditions are described in Figures 1, 2, 3, and 4.
[b] Readily extracted with aqueous acetone according to method of Pons and Guthrie (10). Value for original flakes 0.68%.
[c] Extracted with aqueous acetone after hydrolysis with oxalic acid (11). Value for original flakes 0.69%.
[d] Soluble in 0.5 normal sodium chloride solution.
[e] Dried for 2 hours at 101°C. in forced-draft oven.

involved in oil removal. It remains to be determined whether other factors besides free gossypol content, such as the nature of bound gossypol, affect the nutritive value of the meal for poultry and swine.

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The Detoxication of Tung Meal

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THE tung oil industry of the Gulf coastal area has grown remarkably in the last two decades. The industry produces as by-product large quantities of a press-cake which is quite toxic. This toxicity has restricted the use of the cake to that of a fertilizer or fertilizer extender. The problem of up-grading the cake to a feedstuff has attracted the attention of several laboratories in the area without seemingly producing a clear-cut answer.

Erickson and Brown (4) found a commercial press cake to be non-toxic to rats and reported that oil-free meal prepared in the laboratory could be detoxified by heat alone. Preliminary moistening aided this detoxication. Rusoff, Mehrhof, and McKinney (6) reported that a press cake extracted with naphtha, steamed, and fermented in storage had little, if any, toxicity and small value as a feedstuff. Later Davis, Mehrhof, and McKinney (2) reported that a very toxic press-cake could not be detoxified by heat alone. Chicks were used as experimental animals in both cases. The commercial cakes used by Emmel (3) had a relatively low level of toxicity which decreased during storage. These he converted into non-toxic meals by first extracting with ether and alcohol or acetone and alcohol and then hydrolyzing the residue by autoclaving in the presence of dilute hydrochloric acid. Both solvents were necessary to detoxify the material completely. Emmel concluded that two toxic principles were present: a saponin and a second alcohol soluble material. Chicks were the experimental animals used. In a brief summary devoid of all details Bryan (1) stated that the toxic principle is an albumin and that tung meal may be detoxified quite readily to provide a nutritious feed-stuff for chicks, mice, and cattle

The literature based on research in the Far East does not seem to clarify the above picture. We hope to do so in this report.

Experimental

General Procedure. Two commercial press-cakes were obtained. A solvent extracted meal was also obtained from the Tung Oil Laboratory of the United States Department of Agriculture. This meal had been prepared by cracking and flaking cleaned tung kernels and then extracting the oil without heat by forcing Skellysolve B through the flakes. The final meal was air-dried.

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Preliminary observations indicated that albino rats do not relish diets containing tung products. There were also indications that rats are more resistant to the toxicity than chicks. Chicks readily consumed even the most toxic tung products and showed no resistance to the property. Week-old New Hampshire chicks were accordingly used as test animals. They were fed the rations detailed in Table I. It should be stressed that the experimental ration containing 20% of tung meal is not an economic ration but was designed to give good growth, irrespective of the quality of the tung protein, provided the tung fraction was non-toxic. Extracts of the meals were stripped of solvent and fed in capsules to chicks consuming the stock ration. Feed and water were continually available. Experimental and control groups contained 10-12 chicks unless otherwise specified. The fowls were weighed every fourth day over the experimental period of 24 days.

TABLE I
Composition of Rations

Component	Stock	Experimental
	%	%
Milo	25.0	10.0
Wheat bran	18.0	18.0
Wheat shorts	18.2	18.2
Soybean oil meal	17.0	12.0
Meat scraps	5.0	5.0
Liver meal	2.5	2.5
Alfalfa leaf meal	10.0	10.0
Oyster shell	1.0	1.0
Bone meal	2.0	2.0
Salt	1.0	1.0
Delsterol	0.3	0.3
Tung meal or cake	......	20.0

Toxicity of Meals Used. The cakes and meal were tested as soon as possible after receipt and at intervals thereafter. The results are summarized in Table II. The drop in toxicity of the press-cake is in marked contrast to the stable level of toxicity of the extracted meal. The decrease in toxicity coincided with a change in appearance of the cake from an oily to a dry solid. No further decrease in toxicity was noted thereafter. It seems probable that the initial rapid decrease in toxicity is coupled with oxidation of the residual tung oil, a reaction initiated by the expeller process.

Action of Solvents. Portions of the Skellysolve extracted meal were placed in a Soxhlet type extractor