

TABLE II
Comparative Analysis of Oil Through Various Stages of Plant Processing

	Moisture and volatile	F.F.A.	Loss data chromatographic	Lovibond color 1-in. column crude oil
	%	%	%	
1. Laboratory-extracted oil from cooked meats.....	0.69	0.53	2.88	35/11.3
2. Same as No. 1 plus 15 min. sunlight.....			3.60	35/11.3
3. Same as No. 1 plus sun and air.....			5.08	35/13.6
4. Same as No. 1 plus granular soda ash.....	0.09	0.43	2.19	35/10.6
5. Same as No. 4 plus 15 min. sunlight.....			3.10	35/11.2
6. Same as No. 4 plus sun and air.....			2.48	35/13.6
7. Laboratory-extracted oil from prepressed flake.....	0.80	0.51	3.11	35/13.7
8. Same as No. 7 plus 15 min. sunlight.....		0.50	5.14	35/14.1
9. Same as No. 7 plus sun and air.....		0.56	4.06	35/15.7
10. Plant-prepressed oil low-pressure.....	0.10	0.70	1.33	35/13.3
11. Same as No. 10 plus 15 min. sunlight.....		0.67	3.78	35/15.1
12. Same as No. 10 plus sun and air.....		0.61	4.25	35/18.5
13. Plant-prepressed oil high pressure.....	0.06	0.64	1.34	35/15.7
14. Same as No. 13 plus 15 min. sunlight.....		0.59	4.24	35/22.2
15. Same as No. 13 plus sun and air.....		0.50	2.49	35/26.0
16. Plant solvent-extracted oil.....	0.12	0.53	2.97	35/11.9
17. Same as No. 16 plus 15 min. sunlight.....		0.62	4.63	35/14.9
18. Same as No. 16 plus sun and air.....		0.50	6.36	35/16.6

apparent on oils produced from seed with over 1.0% F.F.A. than is indicated on the 0.5% F.F.A. seed processed during this test period.

A more complete appraisal of cup loss and color data correlated with the rest of the oil analysis shown in Table II is contemplated in the future. We propose to continue this work in our plant along comparably comprehensive lines. Periodic progress reports will be made to the American Oil Chemists' Society when justified by plant and laboratory data obtained.

Summary

Plant operating-procedures and laboratory controls were set up to evaluate the quality of meal and oil which could be produced through each stage of commercial, prepress-solvent-extraction processing. By altering conventional, prepress-solvent-processing conditions and by increasing moisture during cooking and adding granular soda ash after cooking meats, cottonseed meal rations can be produced which are comparable in feed efficiency to soybean meal rations and satisfactory for feeding laying hens in amounts up to 10% of the total weight of the ration with no egg-yolk discoloration and crude cottonseed oils with low F.F.A. and light color can be produced which refined to low Lovibond colors and with refin-

ing losses approximating the chromatographic loss when miscella refined within minutes after separation from the source material with the exclusion of air and light.

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Composition of Acidulated Cottonseed Soapstocks as Influenced by Commercial Methods of Processing Seed and Oil¹

MACK F. STANSBURY, VIDABELLE O. CIRINO, and HAROLD P. PASTOR,
Southern Regional Research Laboratory,² New Orleans, Louisiana

SOAPSTOCKS containing approximately 100 million pounds of anhydrous fatty material are produced as a by-product in refining the annual domestic production of cottonseed oil. The major outlets for this material are as a source of fatty acids and pitch.

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² One of the laboratories of the Southern Utilization Research and Development Division, Agricultural Research Service, U. S. Department of Agriculture.

In recent years increasing amounts of soapstocks have been used as a plasticizer in pelleting oilseed meals and as a source of fat in mixed feeds.

No systematic study of the composition of acidulated soapstocks as related to processing conditions and refining methods has been reported. In fact, relatively little information on their composition is available (3, 8, 9, 13, 20). The trend toward replacement of hydraulic pressing methods with screw-pressing,

TABLE I
 Composition and Properties of Acidulated Cottonseed Soapstocks

Refining process	Refining loss of crude oil	Maximum acidulation temperature	Moisture in acidulated soapstock	Composition and properties—moisture-free basis						
				pH ^a	Total (unoxidized) fatty acids	Neutral oil	Unsaponifiable matter	Oxidized fatty acids	Gossypol	Phosphatide (Total P x 25)
Hydraulic oils										
Centrifugal (10 samples) ^b	%	°F.	%	%	%	%	%	%	%	%
High.....	7.0	240	6.86	6.22	97.3	29.56	6.30	9.28	3.11	2.70
Low.....	3.7	175	1.44	1.88	86.0	2.52	3.45	2.17	0.15	0.53
Mean.....	5.1	198	2.58	3.13	93.3	10.33	4.77	4.27	0.96	1.29
Std. dev. ^g	±1.1	±23.0	±1.54	±1.51	±3.2	±9.24	±0.91	±1.91	±0.97	±0.69
Batch (21 samples) ^c										
High.....	25.6	215	2.78	6.29	95.9	42.53	5.35	4.08	0.93	1.38
Low.....	5.0	190	0.76	1.65	92.2	29.15	1.62	1.41	0.084	0.05
Mean.....	15.4	197	1.38	3.11	94.1	35.67	2.97	2.57	0.46	0.41
Std. dev.	±8.2	±9.6	±0.48	±1.06	±1.0	±3.47	±1.24	±0.69	±0.24	±0.38
Screw-pressed oils										
Centrifugal (9 samples) ^d										
High.....	24.5	220	5.50	6.98	94.2	34.33	6.63	19.38	7.87	3.25
Low.....	2.5	214	1.33	1.67	75.8	4.04	2.86	2.94	0.037	0.25
Mean.....	8.7	216	2.96	3.66	89.4	18.95	4.18	6.41	1.94	1.55
Std. dev.	±6.3	±2.1	±1.44	±2.44	±5.4	±10.82	±1.04	±5.02	±2.53	±0.92
High-speed, screw-pressed oils										
Centrifugal (5 samples)										
High.....	27.4	220	3.07	2.33	93.8	31.93	5.66	7.68	3.93	4.78
Low.....	6.5	216	1.22	1.80	86.2	24.96	2.17	2.13	0.91	2.10
Mean.....	15.3	218	2.29	2.03	90.4	28.99	3.93	4.78	2.73	3.33
Std. dev.	±10.7	±2.2	±0.93	±0.24	±3.0	±2.74	±1.58	±2.50	±1.62	±1.04
Prepress solvent-extracted oils										
Centrifugal (12 samples) ^e										
High.....	7.8	220	12.48	6.05	96.0	31.08	4.50	10.39	4.37	1.88
Low.....	3.1	215	1.05	1.17	86.8	5.71	1.88	2.67	0.07	0.18
Mean.....	5.6	218	3.53	2.32	92.7	16.53	3.64	5.07	0.91	0.62
Std. dev.	±1.4	±2.6	±3.61	±1.25	±2.6	±7.33	±0.80	±2.62	±1.14	±0.48
Miscella (10 samples)										
High.....	12.1	235	30.30	1.79	86.7	40.39	5.45	7.56	1.48	2.75
Low.....	2.7	180	0.38	<0.00	57.7	2.13	2.76	2.65	0.15	0.13
Mean.....	4.3	220	5.04	1.13	80.1	25.01	4.18	5.50	0.66	0.85
Std. dev.	±2.8	±15.0	±9.42	±0.57	±8.4	±11.03	±0.84	±1.42	±0.46	±1.02
Direct-solvent-extracted oils										
Centrifugal (3 samples)										
High.....	5.7	215	33.90	1.93	80.3	14.23	4.60	19.45	6.61	15.75
Low.....	3.7	194	3.40	1.04	67.7	3.30	3.24	5.14	0.441	1.50
Mean.....	4.9	203	22.73	1.36	73.8	7.22	3.88	13.85	3.49	6.88
Miscella (9 samples)										
High.....	5.5	212	39.90	6.67	78.7	3.93	4.14	20.90	10.78	6.58
Low.....	5.2	200	2.40	0.86	67.7	1.93	2.30	16.48	0.84	0.50
Mean.....	5.3	204	20.23	5.00	73.7	2.84	3.19	18.67	3.23	2.68
Std. dev.	±0.15	±6.0	±11.17	±2.20	±3.2	±0.66	±0.76	±1.63	±3.17	±1.74
Blended oils										
Centrifugal (20 samples) ^f										
High.....	21.0	220	6.76	7.20	96.8	34.13	5.71	5.73	1.81	2.23
Low.....	6.0	200	0.78	1.42	90.4	3.80	2.46	2.12	0.013	0.25
Mean.....	9.5	216	2.79	3.57	94.3	8.66	4.00	3.91	0.24	0.70
Std. dev.	±4.4	±6.0	±2.02	±1.91	±1.8	±9.01	±0.95	±1.08	±0.40	±0.46

^a Determined on aqueous extract of soapstock.

^b Refining losses available for only 9 samples.

^c Acidulation temperatures available for only 14 samples.

^d Acidulation temperatures available for only 6 samples.

^e Acidulation temperatures available for only 11 samples.

^f Refining losses available for 13 samples; acidulation temperatures available for 18 samples.

^g Standard deviation.

solvent extraction, and prepress-solvent extraction for processing cottonseed and toward the increased use of centrifugal refining of cottonseed oil has resulted in the production of acidulated soapstocks which vary widely in composition and properties. The present investigation was undertaken to provide information on the variation in composition of commercial acidulated soapstocks to provide for better utilization and for setting standards for trading.

Samples and Methods of Analysis

The 99 acidulated soapstocks analyzed were supplied by 16 commercial refiners and were of known processing histories. They were prepared by commercial or laboratory acidulation of raw soapstocks resulting from centrifugal, miscella, or batch refining, including the caustic soda and soda ash—caustic soda methods. The crude oils from which the samples were derived were obtained from 27 widely located mills

and are representative of the five major processes for the production of cottonseed oil. Data on the processing conditions are summarized in Table I.

The acidulated soapstocks were analyzed for total (unoxidized) and oxidized fatty acids, unsaponifiable matter, and iodine value by use of methods G 3-53, Ca 6b-53, and L 8a-55, respectively, of the American Oil Chemists' Society (1). Moisture was determined by a Karl Fischer-titration procedure adapted from the A.O.C.S. Tentative Method Ca 2e-55 and neutral oil by a modification of the method of Linteris and Handshumaker (11). The colorimetric method of Pons *et al.* (17) was used for determining total phosphorus after a preliminary ashing of the sample with magnesium nitrate. Gossypol was determined by an improved *p*-anisidine colorimetric method (16). The pH of an aqueous extract of the soapstock was measured in order to evaluate the acidity due to mineral acids.

The fatty acid composition of the total (unoxidized) fatty acids was calculated by means of the equations specified in Method Cd 2-38 of the A.O.C.S. from iodine (1) and thiocyanogen (10) values determined on the total fatty acid fraction (unoxidized). The values calculated for saturated acids were corrected to exclude unsaponifiable matter.

Results and Discussion

Composition of Acidulated Soapstocks. The compositional data on a moisture-free basis are summarized in Table I. The samples are grouped according to the type of crude oil from which they were derived and the type of refining process used. The refining losses are caused not only by the free fatty acid content of the crude oil but also by the numerous other factors affecting the emulsification or entrapment of neutral oil and the physical characteristics of the soapstock. Surface-active substances, such as phosphatides, are generally believed to increase refining losses while gossypol has been reported (18) to have a beneficial effect on lowering refining losses. Since these constituents would be present in the acidulated soapstocks, the data obtained were examined to ascertain whether any relationship was apparent between refining loss and the type of cottonseed processing or composition of the acidulated soapstocks. The refining losses averaged highest in the case of the batch-refined, hydraulic oils and the centrifugally-refined, high-speed, screw-pressed oils; and the soapstocks from these oils contained the greatest amounts of neutral oil. The refining losses of the other types of oils were reasonably low, but nevertheless the soapstocks from the screw-pressed oils and from the prepress-solvent oils contained appreciable quantities of neutral oil. There also appears to be no consistent trend between refining loss and either the gossypol or phosphatide content of the samples examined in this study.

The maximum temperature employed in acidulating the soapstocks varied from 175° to 240°F. The effect of variation of acidulation temperature on the over-all composition of soapstocks of a given type was not appreciable. In addition, the samples of a given type which had been laboratory-acidulated were generally comparable in composition to commercial samples.

The moisture contents of most of the soapstocks were in the range of 1 to 3%. However those from the direct-extracted oils were exceptionally high in moisture, averaging 20.8%. Since all of these latter samples were laboratory-acidulated, it seems probable that the high-moisture contents result from inadequacies in the laboratory procedures used. No information is available for commercially acidulated materials from this type of oil. The mineral acidity of the soapstocks varied appreciably as indicated by the pH values of aqueous extracts ranging from almost zero to 7.2. The pH values for a considerable number of the samples were quite low, suggesting incomplete removal of the mineral acid used in the treatment of the raw soapstocks.

The total (free and combined, unoxidized) fatty acid content is an important compositional factor in the commercial evaluation of acidulated soapstocks (12). Of the 99 samples examined, 73 contained 85% or more of total fatty acids and only six contained 95% or more of total fatty acids on the original moisture basis.

The contents of total fatty acids (unoxidized) of the soapstocks derived from the direct-solvent-extracted oils were consistently lower than those from other types. The samples from miscella refined, prepress-solvent oils also averaged somewhat lower in total fatty acids while those from centrifugally refined, prepress-solvent oils contained amounts approximately comparable with those present in the remaining types of samples.

As might be anticipated, higher neutral oil contents were observed for the soapstocks from hydraulic oils which had been batch-refined than for those which had been centrifugally refined. Most of the samples from screw-pressed, high-speed screw-pressed, and prepress-solvent oils contained comparatively large amounts of neutral oil while most of those from direct-extracted and blended oils were low in neutral oil.

The unsaponifiable matter did not vary greatly, and no change attributable to the type of process was observed. This material consists predominantly of sterols, together with smaller amounts of higher aliphatic alcohols, pigments, and other substances which are not saponifiable with alkali but which are soluble in fat solvents. There is apparently some concentration of unsaponifiable matter in the soapstocks since crude cottonseed oils usually contain about 1.6% sterols or less (7), and refined oils of American origin usually contain 0.6–1.0% of unsaponifiable matter (2).

The material determined as oxidized fatty acids includes those substances which are soluble in ethyl ether but insoluble in petroleum ether and therefore remain after isolation of the total fatty acids (unoxidized). This fraction is reported to increase still-pot losses and reduce operating time between cleaning operations in fatty acid distillation plants (8). Analysis of the oxidized fatty-acid fractions from several of the soapstocks indicated the presence of relatively small amounts of phosphatides and gossypol. Modified forms of gossypol resulting from the hot alkaline saponification, which are not measured in the analytical procedure for gossypol, would be expected to be present as well as appreciable quantities of polymerized fatty material.

The samples, with the exception of those from the direct-solvent-extracted oils, contained fairly low amounts of oxidized fatty acids, averaging from 3.12% to 6.41%. Those from the direct-solvent-extracted oils were markedly different in that they contained much larger amounts of oxidized fatty acids, averaging 17.46%. Since mildly heated or tempered flakes, rather than cooked flakes, are generally used in the direct-solvent-extraction process, a considerable amount of non-oil constituents are present in the crude oils (5) and should also be found in the resultant soapstocks. Commercial oils probably contain quantities of oxidized fatty acids intermediate between the values reported by Eaves *et al.* (5) for oils extracted from raw and tempered flakes. If all the oxidized fatty acids in the crude oils were present, the calculated contents in the acidulated soapstocks would range from about 8% for tempered flakes to about 25% for raw flakes. It is interesting to note that the average value of 17.46% found for oxidized fatty acids in soapstocks from direct-solvent-extracted oils is intermediate between these calculated values.

Gossypol was one of the most variable constituents in the acidulated soapstocks, ranging from 0.013 to

TABLE II
Composition of Total (Unoxidized) Fatty Acids of Acidulated Cottonseed Soapstocks

Refining process	Iodine value (Wijs) of dry, filtered soapstock	Composition—moisture-free basis					
		Iodine value (Wijs)	Thiocyanogen value	Linoleic acid	Oleic acid	Saturated acids	Unsapo- nifiable matter ^a
				%	%	%	%
Hydraulic oils							
Centrifugal (10 samples)							
High.....	106.8	104.5	63.3	48.73	22.09	30.39	6.64
Low.....	99.2	98.2	61.1	43.54	17.71	27.70	3.69
Mean.....	102.8	101.5	62.3	46.28	19.66	28.94	5.12
Std. dev.....	±2.7	±2.3	±0.8	±1.93	±1.46	±0.86	±0.96
Batch (21 samples)							
High.....	107.8	109.4	66.3	51.75	19.61	31.93	5.80
Low.....	102.0	102.4	62.5	47.14	14.70	27.01	1.69
Mean.....	105.6	106.5	64.2	50.07	17.62	29.15	3.16
Std. dev.....	±1.5	±1.4	±0.8	±1.24	±1.52	±1.50	±1.33
Screw-pressed oils							
Centrifugal (9 samples)							
High.....	108.1	109.5	64.7	52.93	20.84	30.80	7.37
Low.....	99.2	98.8	60.8	44.89	13.32	26.92	3.04
Mean.....	103.5	104.5	63.3	48.68	18.20	28.43	4.70
Std. dev.....	±2.8	±2.9	±1.3	±2.47	±2.41	±1.15	±1.19
High-speed, screw-pressed oils							
Centrifugal (5 samples)							
High.....	108.2	107.2	65.6	49.89	20.42	28.34	6.57
Low.....	105.1	104.2	62.6	48.02	16.05	26.56	2.31
Mean.....	106.3	105.7	64.3	48.98	18.93	27.70	4.39
Std. dev.....	±1.5	±1.3	±1.3	±0.68	±1.72	±0.71	±1.88
Prepress solvent-extracted oils							
Centrifugal (12 samples)							
High.....	109.2	106.4	63.7	50.46	28.75	45.61	4.90
Low.....	69.0	68.5	48.5	23.54	15.90	28.50	2.10
Mean.....	100.0	99.0	60.9	44.96	19.52	31.62	3.91
Std. dev.....	±10.1	±10.0	±4.2	±7.05	±3.51	±4.59	±0.82
Miscella (10 samples)							
High.....	100.5	108.8	65.5	51.67	20.63	37.37	9.38
Low.....	95.0	79.6	50.2	34.76	14.03	26.66	3.68
Mean.....	97.8	99.2	60.4	45.95	17.78	30.94	5.33
Std. dev.....	±2.0	±10.4	±5.1	±6.51	±2.14	±4.22	±1.62
Direct solvent-extracted oils							
Centrifugal (3 samples)							
High.....	107.5	109.8	64.8	53.75	20.46	28.78	6.26
Low.....	82.2 ^b	102.7	63.1	46.54	15.83	25.63	4.74
Mean.....	92.9	106.0	63.7	50.10	17.59	27.05	5.26
Miscella (9 samples)							
High.....	115.8	112.0	66.9	54.58	23.02	28.68	5.72
Low.....	88.6 ^b	102.5	64.3	45.16	14.61	24.45	3.14
Mean.....	100.1	109.5	65.7	51.77	17.47	26.43	4.33
Std. dev.....	±9.2	±2.8	±0.9	±2.88	±2.96	±1.30	±1.03
Blended oils							
Centrifugal (20 samples)							
High.....	111.7	108.8	65.6	53.27	27.73	31.95	6.07
Low.....	99.2	99.8	60.9	42.21	12.93	26.83	2.62
Mean.....	105.7	105.3	63.9	48.97	18.46	28.32	4.25
Std. dev.....	±3.8	±2.7	±1.4	±3.05	3.70	±1.30	±1.02

^a Calculated from % unsaponifiable matter and % total fatty acids in acidulated soapstock.

^b Filtration not feasible; analyses on vacuum oven-dried, unfiltered sample.

10.78%. The samples from direct-solvent-extracted oils were highest in gossypol content, averaging 3.30%. This would be expected since crude oils obtained by this process usually contain more gossypol than those produced by other commercial processes (4, 14, 15). All of the soapstocks from the high-speed, screw-pressed oils were also relatively high in gossypol while both high and low gossypol contents were observed in soapstocks from screw-pressed and prepressed-solvent oils. The soapstock from the hydraulic oils averaged 0.62% gossypol, which is lower than any other type except those from blended oils, which averaged 0.24%. The fact that some of the latter soapstock samples had been purified by treatment with alkali and washing prior to acidulation may account in part for their low gossypol values.

Although gossypol tends to concentrate in the soapstocks, the amounts found are somewhat lower than would be expected if all of the gossypol in the crude oils was removed with the soapstocks. This is undoubtedly caused by the modification or destruction of some of the gossypol during the refining and acidulation processes. In this connection the gossypol present in the acidulated samples is apparently

not "bound" since analysis of 32 representative soapstocks for total gossypol, using an acid hydrolysis procedure (16), gave values in good agreement with those obtained without acid hydrolysis.

Variations in the phosphatide content of the various types of soapstocks followed the same general pattern observed for gossypol. This is reasonable as the quantities of both gossypol and phosphatides in crude cottonseed oils are related in a similar manner to the methods of cooking and oil extraction used. The samples from direct-extracted and high-speed, screw-pressed oils were high in phosphatides. Those from screw-pressed oils were intermediate; and the hydraulic, prepress-solvent, and blended types were somewhat lower. Although the reported values for phosphatides were calculated from the total phosphorus values, it is felt that they are good estimates. This is supported by the fact that total nitrogen values determined on a number of representative samples ranged from 0.10 to 0.44% and ranked in the same order as the phosphorus contents. The ratios of phosphorus to nitrogen were of the order of magnitude expected for phosphatides. There was no consistent relationship between moisture content of the

acidulated soapstocks and their phosphatide content.

Composition of Total Unoxidized Fatty Acids. The composition of the total fatty acids (unoxidized) in the soapstocks is summarized in Table II. For most of the samples, except those from direct-extracted oils, the iodine values were approximately the same as those for the dry, filtered soapstocks. The differences in the case of samples derived from the direct-extracted oils probably result from the high oxidized fatty-acid content. Thus it would be possible to predict roughly the degree of total unsaturation of the fatty acids from the iodine value of the soapstock. The average iodine values observed for the total fatty acid fractions (unoxidized) ranged from a low of 99.1 for those derived from prepress-solvent oils to a high of 108.6 for those from direct-solvent oils. These two types also averaged lowest and highest in thiocyanogen values. Although only limited data are available on the iodine values of the crude oils which were refined to produce the soapstocks used, these data indicate that the iodine values of the total fatty acids from the soapstocks average approximately five units lower than the corresponding iodine value of the crude oil. This trend has recently been observed by others for several vegetable oils (6) and is borne out by the fatty acid composition data in Table II. It appears that there is a decrease in the proportional amount of unsaturated fatty acids with a corresponding proportional increase in saturated fatty acids as a result of the refining and/or acidulation.

The average linoleic acid content ranged from 45.51% to 51.35%. These values are in the same general range found for most cottonseed oils (19). Some of the individual linoleic acid contents were slightly higher and others slightly lower than the values reported for cottonseed oils of comparable iodine values. However in almost all cases the oleic-acid contents averaged somewhat lower and saturated acids somewhat higher than normally found for cottonseed oils of comparable iodine values. The total fatty acids from the samples derived from direct-extracted oils contained on the average the lowest amounts of oleic and saturated acids while those from prepress-solvent oils averaged highest in saturated acids, among the highest in oleic acid, and lowest in linoleic acid. The reasonably small standard deviations indicate that the linoleic, oleic, and saturated acid contents in the total fatty-acid fraction (unoxidized) from most soapstocks of a given type varied within relatively narrow limits.

There are definite questions as to the applicability of the iodine-thiocyanogen method for calculating the composition of the fatty acids isolated from soapstock. The presence of octadecadienoic acids, which absorb more thiocyanogen than normal linoleic acid, would lead to high values for linoleic and saturated acids and low values for oleic acid. On the other hand, the spectrophotometric method is also inapplicable in the presence of these isolinoleic acids as they are not conjugated by the alkaline treatment, hence low values for octadecadienoic and saturated acids and high values for oleic acid would be obtained. Analysis of several typical samples by the A.O.C.S. spectrophotometric procedure (1) did indeed give results which were not in accord with those calculated from the iodine and thiocyanogen values. Analysis of these same samples for saturated acids by low temperature crystallization from acetone (4a)

gave values intermediate between those calculated by the other two procedures. The discrepancies observed are what would be expected if a small amount of linoleic acid had been converted to isomeric acids. Examination of the infrared absorption spectra of these samples revealed the presence of approximately the amounts of *trans* acids required and strongly suggests that some isomerization occurred during the production of the soapstock or the isolation of the total fatty acids from them. However it is felt that the fatty-acid composition calculated from the iodine and thiocyanogen values should be useful at least for comparative purposes.

The amount of unsaponifiable matter in the isolated fatty acids was calculated from the percentage of unsaponifiable in the acidulated soapstock and their total fatty-acid content. Although the unsaponifiables in the total fatty acids varied from 1.69 to 9.38%, most of the individual values fell within the narrow range of 2 to 5%. In general, comparable amounts of unsaponifiables were present in the total fatty-acids fraction from soapstocks derived from the various types of crude oils.

Summary

Acidulated cottonseed soapstocks representative of domestic production have been analyzed to obtain compositional data needed for efficient utilization of these materials and for their evaluation in commercial trading. The 99 soapstocks used were of known processing histories and are representative of the five major processes for the production of crude cottonseed oil; of centrifugal, miscella, and batch refining; and of commercial or laboratory acidulations.

Most of the compositional characteristics of the acidulated soapstocks were quite variable. The soapstocks from direct-solvent-extracted oils contained the least total fatty acids (unoxidized) and the most oxidized fatty acids. The samples from hydraulic oils averaged highest in total fatty acids and lowest in oxidized fatty acids. Those from oils produced by the three remaining processes did not differ greatly from the samples derived from hydraulic oils with respect to these two constituents. High neutral-oil contents were observed consistently for the soapstocks from hydraulic crude oils which had been batch-refined and from high-speed, screw-pressed oils which had been centrifugally refined. Most of the samples from screw-pressed and prepress-solvent oils also contained relatively large amounts of neutral oil while most of those from direct-extracted and blended oils were low in this constituent. Gossypol was one of the most variable constituents. The samples from the direct-extracted and high-speed, screw-pressed oils were comparatively high in both gossypol and phosphatides whereas those from hydraulic oils were quite low in each of these constituents.

The composition of the total fatty acids (unoxidized) from the soapstocks was in the same general range as usually found for cottonseed oils, with the exception that the oleic-acid contents averaged somewhat lower and the saturated acids somewhat higher. These differences may result in part from the presence of small amounts of isolinoleic acids. Relatively small differences in composition of total fatty acids were found as related to methods of cottonseed processing.

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Determination of Tocopherol in Oxidized Fats¹

E. N. FRANKEL, C. D. EVANS, and J. C. COWAN, Northern Utilization Research and Development Division, Agricultural Research Service, U. S. Department of Agriculture, Peoria, Illinois

SEVERAL MODIFICATIONS of the Emmerie-Engel method (2) for the determination of tocopherol in fats have been developed to remove substances which interfere with the ferrous-bipyridine color reaction (1, 2, 7, 8). Fats were reported to interfere with this color reaction (4, 6). Kaunitz and Beaver (6) introduced a proportionality factor to correct for the color-depressing effect observed in the presence of increasing concentrations of fat. However Gupta and Basu (3) found that this correction factor did not apply to oxidized groundnut oil. They showed that this oil when free of peroxides did not inhibit the color development in the Emmerie-Engel procedure. Therefore this interference of fats with the color reaction may be caused by peroxides in the fats. Lips (7) used the sulfuric-acid treatment of Parker and McFarlane (9) and saponification at room temperature to remove interfering substances in autoxidized methyl esters of fatty acids.

This paper describes a simple method for tocopherol determination in oxidized fats where peroxides are removed by thermal destruction since the presence of peroxides gives erroneously low values. The method is currently being used in a study of the fate of tocopherols in oxidizing fats.

Experimental

The method of Stern and Baxter (11) was used for tocopherol, except 10 min. were allowed for color development instead of 2.5 min. Preliminary obser-

vations showed that soybean oil immediately after deodorization at 210°C. did not interfere with the Emmerie-Engel color reaction for tocopherol. When a series of determinations was carried out with different concentrations of freshly deodorized oil (20 to 140 mg. per 10-ml. solution), the color developed was proportional to the sample weight. However, when allowed to oxidize, the oils interfered with the determination of tocopherol (Figure 1). These results are in agreement with those of Gupta and Basu (3) in showing the interference of fat peroxides with the Emmerie-Engel color reaction for tocopherol.

A study was made of the effect of deodorization on the tocopherol and peroxide contents of soybean oil and lard. The fats were heated at 210°C. under reduced pressure (less than 1 mm. Hg.) in 50-ml., round-bottom flasks immersed in a thermostatically

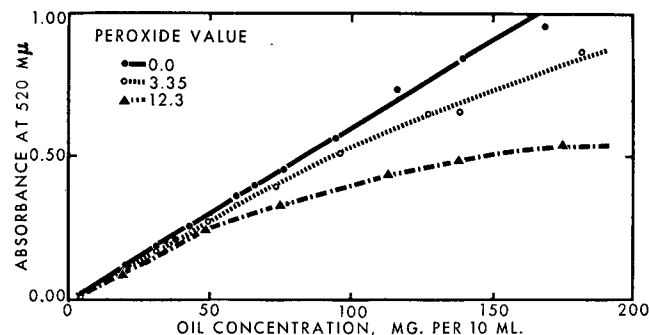


FIG. 1. Effect of peroxides in soybean oil on the ferrous-bipyridine color reaction for tocopherol.

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