Suitability of Commercial Cottonseed for Producing Edible High Protein Flours by Liquid Classification

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ABSTRACT

The 1976 and 1977 crops of glanded cottonseed from oil mills located in 7 major U.S. cottonbelt growing areas were evaluated as to their suitability for processing into edible, high-protein flours. Both the physical and chemical characteristics of fuzzy seed samples were studied. Ginned seed samples were hulled, dried, comminuted, slurried with hexane and liquid classified by both a laboratory differential settling test (DST) and pilot plant liquid cyclone process (LCP). Liquid cyclone operating data and extensive analytical data of the kernels, flour and meals produced are shown. A free gossypol level of 1.10% or less in full fat kernels was the determining factor for producing edible flours from glanded seed by the LCP. The Lower Rio Grande Valley area was the only area from which liquid classified cottonseed flour consistently exceeded the current maximal free gossypol standard of 450 ppm. Ca. 94% of the total U.S. cottonseed production is suitable for producing edible, high-protein flours by liquid classification.

INTRODUCTION

Although cottonseed represents roughly 10% of the total value of a cotton crop, it is an essential segment of the farmer's monetary return. Cottonseed is normally processed into oil, meal, hulls and linters, with most of the value in the oil and meal. With the recent sharp increase in sunflower oil production (1) and the anticipated huge increase in distillers' dried grain plus solubles supplies (a ruminant feed byproduct from fuel alcohol production) over the next 10 years (2), additional market areas for cottonseed products will be needed if the crop is to prosper. One of these areas might be in providing protein for the nation's food supply. The laboratory development of the liquid cyclone (3-5) and air classification (6) processes, and the development of glandless (very low gossypol) seed, have made edible cottonseed protein supplies available for research. Researchers have responded by demonstrating the uniqueness of the chemical and nutritive properties of cottonseed protein and its potential uses which could provide an important future outlet for cottonseed (7-11).

In order to develop and maintain a market for an edible product, a large and reliable supply of cottonseed protein is necessary. Glandless cottonseed production, although increasing, is still insufficient to supply the amounts needed. The liquid cyclone process (LCP) has this potential. However, during its development, Gardner et al. (4) determined that cottonseed produced on the Texas High Plains processed differently from cottonseed produced in the Mississippi Delta region. The High Plains seed, when processed through roll flaking and liquid cyclone operations, experienced excessive gland rupture and released a myriad of 0.1-1.0 μ pigment-containing spherules that could not be separated by a standard 7.6-cm (3-in.) diameter cyclone (12). It was postulated that the processing difference stemmed from the facts that cottonseed from the High Plains contained both significantly less moisture and (because of the short growing season) a greater amount of immature seed than did the Mississippi Delta-produced cottonseed. Both seed samples were of prime grade and quality, as determined by the cottonseed trading rule factors (13) (percentage of oil, ammonia, moisture, foreign matter and free fatty acids in the oil). However, these factors, adequate for predicting the production of high-grade oil and feed meal products for oil mill operations, are not definitive enough to determine if a specific lot of cottonseed can be used in the LCP to produce an edible protein concentrate.

The negative processing difference was overcome by Gardner et al. (4) who modified the LCP by substituting a dry pin milling operation in place of roll flaking and wet stone milling. This change not only produced edible products from both Delta and High Plains seed, but also simplified the process and yielded higher flour recoveries. To prove the general applicability and viability of the LCP, it was necessary to determine the liquid cyclone processibility of cottonseed produced throughout the growing belt.

Past laboratory liquid classification studies using informal differential settling techniques (14) on cottonseed grown throughout the U.S. cottonbelt have shown that cultivars, growing locations, crop year and/or their interaction terms are highly significant sources of variation associated with the quality of classified products (15,16). Although this information may be of value to breeders and growers, it is of little value to oil mills because it would be impractical to segregate cultivars at gin or mill sites for specific liquid cyclone processing. Because of these impracticalities, this study was undertaken with mill run seed that represented the average grade of seed received for the particular crop year and that were free of Salmonella and aflatoxins.

EXPERIMENTAL PROCEDURES

Materials

One-ton lots of delintered seed and 11.3-k (25-lb) samples of their initial ginned seed base were obtained from oil mills in 7 major U.S. cottonbelt growing areas for the 1976 and 1977 growing seasons. The cottonseed growing areas shown in Figure 1 included California (CA), Southwest Texas (SWTX), Texas High Plains (TXHP), Lower Rio Grande Valley (LRGV), Central East Texas (CETX), Mississippi Delta (MSD), and the Carolina-Georgia region (C-GA). The Arizona-Southern California area was omitted due to a normally high incidence of aflatoxin contamination. The solvent used was Skellysolve B, a commercially available hexane with a high content of N-hexane.

Methods

Hulling. All delintered seed samples were hulled in the pilot plant to yield a whole and cracked kernel fraction containing less than 3% hulls.

Drying. Whole and cracked kernel fractions were dried on a pilot plant continuous belt dryer using air at 82 C (180 F). All kernel samples were dried to below 2.0% moisture.

Grinding. For the differential settling test (DST), dried kernel samples were comminuted through an Alpine 160 Z laboratory model Kolloplex Pin Mill operated at a speed of 9,300 rpm. For the pilot LCP tests, comminution was

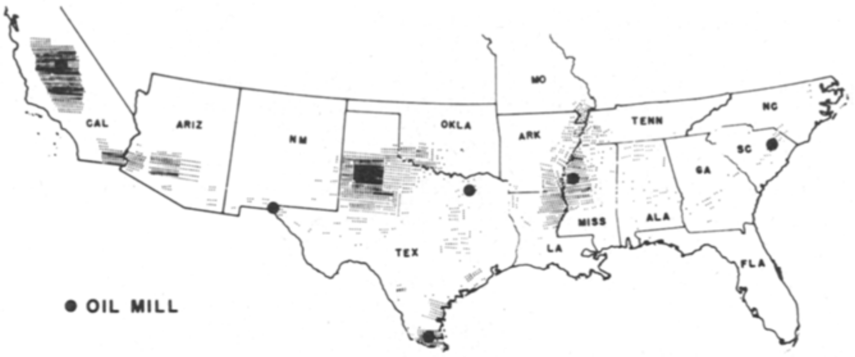

FIG. 1. Cotton producing areas in the United States and oil mill sampling sites.

performed through a commercial 250 CW Alpine Contraplex Pin Mill operated at a door pin disc speed of 2,500 rpm and mill-side disc speed of 7,350 rpm for all samples except those from LRGV and C-Ga. For the latter 2 samples, the mill-side disc speed was reduced to 5,400 rpm to minimize gland rupture and obtain flours with edible free gossypol levels.

Differential settling test. The procedure used for the DST was as described by Hron (17). It consisted of allowing mixed slurry samples, (hexane and 100 g of pin milled kernels), to settle for 20 min in a 1,000-mL graduated glass stoppered cylinder. Before siphoning the supernatant containing the suspended flour solids and representing liquid cyclone overflow (OF). Additional hexane was added to the settled solids, representing the liquid cyclone underflow (UF), to bring the total volume to 1,000 mL, and the above procedure was repeated. After 4 settlings, the composite OF (flour) and UF (meal) solids were recovered by filtering the slurries on a Buchner funnel, washing the resulting cakes, then desolventizing in ambient air overnight and in a forced-draft oven for 1 hr at 101 C (214 F). All tests were performed in duplicate.

Liquid cyclone process. LCP data were obtained by feeding 18% solids slurries containing 36-Kg (80-lb) batches of pin-milled, full-fat kernels and hexane into the pilot plant liquid cyclone at a pressure of 137 kPa (20 psig). The process apparatus is shown in Figure 2. The cyclone used in the tests was a 7.6-cm (3-in.) diameter commercial Demco low-pressure stainless steel cone modified by restricting the feed inlet to 2.5 cm (1-1/2 in.) id, and the overflow (vortex finder) and underflow (apex) outlets to 1.6 cm (5/8 in.) and 2.5 cm (1/2 in.) id, respectively. The modifications reduced throughput to quantities that could be efficiently handled in the pilot plant to give significant data. The pressurized feed slurry was separated or split by the cyclone into an OF slurry fraction containing high-protein flour solids, and a high-gossypol coarse meal UF slurry fraction. Cyclone split was controlled by adjusting the speed of the positive displacement UF pump. Two test runs at both 450 and 480 rpm UF pump speeds were made on milled kernel samples from each of the 7 growing areas for 2 crop years to obtain ranges of comparable liquid cyclone flour recovery and gossypol gland separation efficiency data. The resulting OF and UF slurry streams were recycled to the feed tank, and samples were taken for materials balance calculations and laboratory analyses. After sampling for material balance data, the total batch was processed through the cyclone and the resulting steams were collected in stainless steel drum receivers. The batches were replicated and the resulting OF slurries were combined, filtered and washed with hexane on a rotary vacuum drum filter,

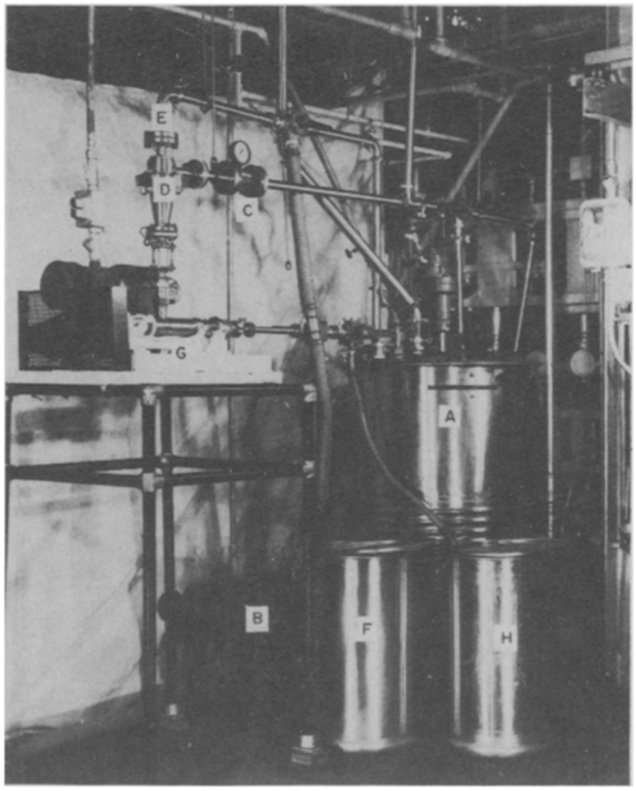

FIG. 2. Pilot plant liquid cyclone process apparatus. (A) Agitated feed tank, (B) feed pump, (C) feed pressure gauge, (D) liquid cyclone, (E) overflow fraction, (F) overflow receiver, (G) underflow pump, (H) underflow receiver.

desolventized overnight at room temperature and then for 2 hr at 93 C (200 F) in a forced-draft oven.

Analytical measurements. Moisture, free gossypol, nitrogen, lipids, free fatty acids, crude fiber, and ash were determined by standard AOCS methods (18). Total gossypol was determined by a modified AOCS method using 3-amino-1-propanol as described by Pons et al. (19).

RESULTS

Table 1 shows mean recovery and analytical data of replicate processing of 2 crop year kernels from 7 growing areas through the laboratory DST and pilot plant LCP at an UF pump speed of 480 rpm. The highest LCP flour recovery was obtained from SWTX kernels (49.3%) and the lowest from C-GA kernels (38.7%). The low recoveries from

TABLE I

Analytical Data on Cottonseed Kernels and Liquid Classified Products[a]

Growing Area	Growing Year	Sample description	Flour or meal recovery (%)[b]	Gossypol Free (%)	Gossypol Total (%)	Protein (%)[c]	Fiber (%)	Ash (%)
CA	1976	Kernels	–	1.04	1.14	61.6	1.8	4.9
		DST[d]-flour	23.0	0.038	0.110	68.7	2.3	7.8
		LCP[e]-overflow flour	42.6	0.041	0.164	68.1	2.3	7.6
		Underflow meal	57.4	2.22	2.36	56.0	3.1	6.3
	1977	Kernels	–	0.940	1.06	59.1	2.1	4.7
		DST-flour	15.1	0.019	0.053	67.2	1.8	7.7
		LCP-overflow flour	45.8	0.037	0.186	66.2	1.9	8.1
		Underflow meal	54.2	2.11	2.91	56.9	3.2	6.9
SWTX	1976	Kernels	–	1.14	1.18	57.9	3.2	4.9
		DST-flour	21.3	0.046	0.971	64.5	2.2	8.4
		LCP-overflow flour	44.6	0.048	0.242	66.1	2.3	8.5
		Underflow meal	55.4	2.37	3.42	53.0	3.0	6.8
	1977	Kernels	–	0.966	1.04	58.8	1.8	5.0
		DST-flour	35.9	0.026	0.038	66.4	2.4	8.7
		LCP-overflow flour	49.3	0.033	0.188	65.7	2.3	8.2
		Underflow meal	50.7	2.66	3.34	53.9	3.2	7.1
TXHP	1976	Kernels	–	0.979	1.05	57.0	2.6	4.6
		DST-flour	33.8	0.038	0.061	67.7	2.5	7.8
		LCP-overflow flour	42.3	0.041	0.173	66.4	3.3	7.4
		Underflow meal	57.7	2.40	2.59	51.4	5.7	7.3
	1977	Kernels	–	1.03	1.19	58.7	0.7	4.3
		DST-flour	23.4	0.021	0.042	64.8	0.8	7.1
		LCP-overflow flour	48.7	0.038	0.165	64.2	0.7	7.0
		Underflow meal	51.3	2.45	2.82	51.8	1.3	6.0
LRGV	1976	Kernels	–	1.34	2.26	59.1	2.0	5.0
		DST-flour	22.5	0.060	0.115	65.4	2.6	8.0
		LCP-overflow flour	42.4	0.064	0.260	65.3	2.2	8.2
		Underflow meal	57.6	2.84	5.92	52.6	3.5	6.7
	1977	Kernels	–	1.32	1.36	63.9	1.0	5.0
		Kolloplex DST-flour	16.8	0.054	0.081	63.8	1.5	8.2
		LCP-overflow flour	39.2	0.060	0.225	66.1	–	8.5
		Underflow meal	60.8	3.90	3.91	54.3	2.0	6.7
CETX	1976	Kernels	–	0.794	1.53	62.4	2.1	4.8
		DST-flour	21.4	0.013	0.034	67.0	2.3	7.6
		LCP-overflow flour	44.7	0.023	0.114	66.4	2.2	7.6
		Underflow meal	55.3	1.71	1.88	55.0	3.5	6.3
	1977	Kernels	–	0.768	1.00	61.7	4.0	4.9
		DST-flour	19.8	0.021	0.045	66.0	2.3	7.5
		LCP-overflow flour	45.0	0.028	0.132	66.1	2.3	7.7
		Underflow meal	55.0	3.00	3.04	56.0	3.3	6.6
MSD	1976	Kernels	–	1.09	1.18	60.2	3.0	5.1
		DST-flour	19.6	0.027	0.043	67.0	2.7	7.9
		LCP-overflow flour	42.1	0.041	0.163	66.3	2.2	8.0
		Underflow meal	57.9	2.54	2.80	58.1	2.8	7.0
	1977	Kernels	–	1.08	1.32	61.1	2.4	5.4
		DST-flour	30.4	0.041	0.156	66.8	2.3	8.2
		LCP-overflow flour	43.7	0.043	0.184	67.4	1.9	8.4
		Underflow meal	56.3	2.71	3.24	57.7	3.2	6.9
C-GA	1976	Kernels	–	1.07	1.19	62.6	3.1	4.9
		DST-flour	24.2	0.021	0.051	67.8	2.2	8.3
		LCP-overflow flour	45.5	0.040	0.138	68.5	2.6	8.0
		Underflow meal	54.5	2.90	3.34	56.1	3.0	6.8
	1977	Kernels	–	1.05	1.30	62.3	4.1	5.0
		DST-flour	28.6	0.044	0.170	68.5	2.4	8.3
		LCP-overflow flour	38.7	0.042	0.226	68.6	2.3	8.3
		Underflow meal	61.3	2.56	3.09	56.0	4.0	6.9

[a]As-is-basis.
[b]Weight-percent basis.
[c]Moisture-/and oil-free basis, N × 6.25.
[d]Differential settling test.
[e]Liquid cyclone process.

C-GA and the LRGV (39.2%) cottonseeds for the 1977 crop year were attributed to the slower speed of the Contraplex pin mill required to minimize pigment gland rupture. Although DST flour recoveries were not directly indicative of pilot plant results, linear regression analysis of the free gossypol (FG) values of the DST and LCP flours gave a straight line relationship (Fig. 3) with a coefficient of variation of 0.898. Further evaluation of FG data in Table I shows that FDA-approved edible levels, (equal to or less than 0.045%) (20), usually were obtained from all the growing areas except LRGV.

The FG content of a few LCP flour samples produced from kernels grown in other growing areas exceeded 0.045% (Fig. 4). However, cottonseed grown in the LRGV area always produced flours containing high FG in the DST (0.060%, 0.054%) and LCP (0.064%, 0.060%) tests. Figure 4 shows the range of values obtained from 8 instantaneous samples taken during 4 LCP runs on each area's seed

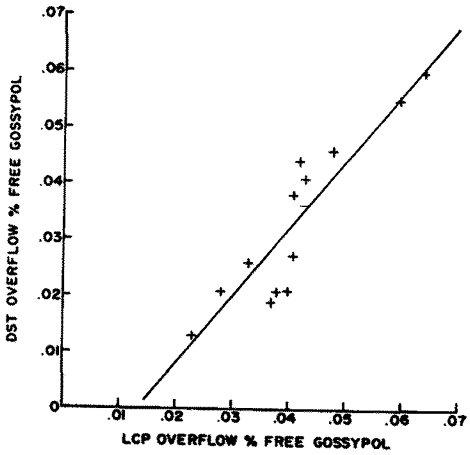

FIG. 3. Comparison of gossypol classification in laboratory DST vs pilot plant LCP.

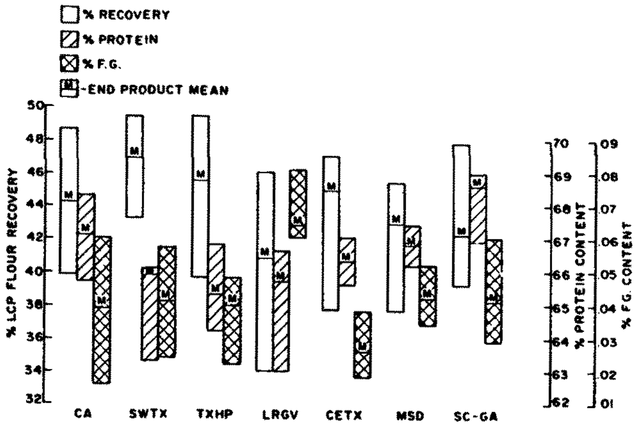

FIG. 4. Range of values for LCP flour: recovery, protein and FG content by growing area.

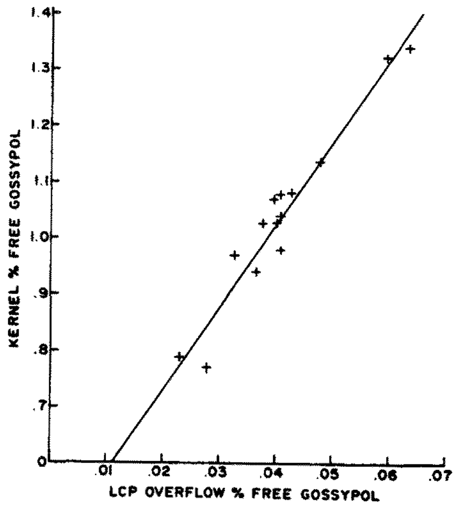

FIG. 5. Relationship of FG content in cottonseed kernels and LCP flours.

sample. Indicated in each bar showing the range of values is the mean value. Although no one seed area was outstanding in all 3 of the variables surveyed, the large ranges show considerable seed variability and latitude in operating values obtainable from the liquid cyclone.

A least square plot of FG contents of kernel and corresponding LCP flour, shown in Figure 5, is a straight line with a coefficient of variation of 0.970. Based on this relationship, kernel FG contents of 1.10% or less will consistently yield LCP flours with FG content below 0.045%.

Kernel protein content, on a moisture and oil free base as shown in Table I, ranged from a low of 57.0% for the TXHP area to a high of 63.9% for the LRGV area. Liquid classification of pin-milled kernel and hexane slurries in the LCP resulted in flours having increased protein contents ranging from a low of 64.2% for TXHP to a high of 68.6% for C-GA. LCP product fiber content values ranged from 0.7 to 3.3% in the flour and from 1.3 to 5.7% in the meal fractions, again showing the classification ability of the liquid cyclone. Ash or mineral content of the LCP flours also showed classifying effects by the cyclone varying from 6.0 to 7.1%.

Kernel lipids and free fatty acid content data, although ranging from 32.1 to 38.6% and 0.3 to 3.8%, respectively, were not included in Table I because a relationship with processibility, i.e., percentage flour recovery, FG content, or classification, could not be determined.

LCP pilot plant operating data in Table II show the individual processing variability of each area's seed under constant operating conditions, i.e., fixed feed and underflow rates. The average FG contents of 28 total LCP static overflow test samples taken on all 14 seed samples at both 450 and 480 rpm underflow pump speeds were 0.045% and 0.041%, respectively, and confirms the cyclone's consistent pigment gland classification efficiency. The data, representing ca. 1/3 of the cyclone's maximal capacity rate, can be easily and reliably scaled-up for designing an LCP plant within the U.S. cotton growing belt.

Normal grade evaluation tests on fuzzy (undelintered) seed samples, (Table III) exhibited wide variations. Although both CETX and MSD area seed were consistently below prime (100%) grade, they processed satisfactorily through the LCP, indicating that grade does not correlate well with LCP flour recovery, protein and FG content. Free fatty acid (FFA) contents varied from 0.4 to 4.2%, but did not affect processing ability. The possible effects of the FFA content on the taste of LCP flours was not evaluated. Extensive examination of fuzzy seeds indicated that physical characteristics such as seed damage, maturity, and pigment gland size, number and fragility varied widely, but had little relationship with processing ability.

Although many characteristics varied, free gossypol content was the controlling factor in determining the liquid cyclone processing ability of commercial seeds obtained from 7 oil mills located within the major U.S. cottonseed growing belt. This study shows that hulled cottonseed kernels containing FG levels of ≤ 1.10%, when processed through the LCP, yield an edible, high-protein flour. Although LRGV was the only area to consistently exceed the limiting FG level of 1.10%, over 94% of the total U.S. production of cottonseed is below this limit. Therefore, the LCP can provide a valuable product that could strengthen the economic position of the cottonseed industry and help feed a hungry world.

ACKNOWLEDGMENTS

The authors thank L.M. Molaison for free and total gossypol analyses and the organizations that provided seed samples and/or cooperation.

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TABLE II

Pilot Plant Liquid Cyclone Processing Data

Growing area	Underflow pump speed (rpm)	Solids feed conc. (%)	Feed rate (lb/min)	Overflow rate (lb/min)	Underflow rate (lb/min)	Overflow solids (%)	Underflow solids (%)	Cyclone split[a] Slurry Overflow (%)	Cyclone split[a] Slurry Underflow (%)	Cyclone split[a] Solids Overflow (%)	Cyclone split[a] Solids Underflow (%)
CA	450	17.6	81.2	61.2	20.0	10.6	39.0	75.4	24.6	45.4	54.6
	480	17.7	81.3	60.1	21.2	9.9	39.7	73.9	26.1	41.4	58.6
SWTX	450	17.0	82.1	62.2	19.9	10.4	37.5	75.8	24.2	46.6	53.4
	480	17.0	82.3	61.2	21.1	10.1	37.0	74.4	25.6	44.2	55.8
TXHP	450	18.3	81.5	61.1	20.4	10.8	40.8	75.0	25.0	44.2	55.8
	480	18.5	80.9	59.3	21.6	10.4	40.6	73.9	26.7	41.2	58.8
LRGV	450	18.1	82.5	61.9	20.5	10.3	41.8	75.1	24.9	42.6	57.4
	480	18.1	83.3	61.9	21.4	9.7	42.4	74.3	25.7	39.8	60.2
CETX	450	18.4	82.9	62.5	20.5	10.7	41.9	75.3	24.7	43.9	56.1
	480	18.4	83.2	61.8	21.5	10.3	41.8	74.2	25.8	41.5	58.5
MSD	450	18.7	84.1	63.3	20.8	10.8	42.5	75.3	24.7	43.7	56.3
	480	18.4	84.5	62.3	22.2	9.6	43.0	73.7	26.3	38.6	61.4
C-GA	450	17.7	84.0	63.4	20.6	10.5	39.8	75.5	24.5	44.8	55.2
	480	17.7	83.4	61.6	21.8	9.9	39.6	73.8	26.2	41.4	58.6
Mean[b]	450	18.0	82.6	62.2	20.4	10.6	40.5	75.3	24.7	44.5	55.5
		± 0.5	± 1.1	± 0.8	± 0.3	± 0.2	± 1.7	± 0.2	± 0.2	± 1.2	± 1.2
	480	18.0	82.7	61.2	21.5	10.0	40.6	74.0	26.1	41.2	58.8
		± 0.5	± 1.2	± 1.0	± 0.3	± 0.3	± 1.9	± 0.2	± 0.4	± 1.6	± 1.6

[a]Weight % basis.
[b]Mean values ± standard deviation.

TABLE III

Fuzzy (Undelintered) Cottonseed Analyses

Growing Area	Growing Year	Oil (%)	Free fatty acids (%)	Protein (%)[a]	Quantity index	Grade
CA	1976	19.8	0.7	21.8	109.6	109.5
	1977	17.9	0.4	21.6	101.7	101.5
SWTX	1976	20.9	0.5	18.9	110.7	110.5
	1977	19.0	0.7	20.3	104.6	104.5
TXHP	1976	18.7	0.7	19.6	102.6	100.5
	1977	19.2	0.5	20.9	106.2	106.0
LRGV	1976	18.3	2.3	19.1	100.5	98.5
	1977	18.7	1.0	19.0	101.9	102.0
CETX	1976	17.4	0.4	20.8	98.9	99.0
	1977	16.0	1.0	20.8	93.2	93.0
MSD	1976	17.1	0.4	19.8	96.4	96.5
	1977	18.0	6.0	22.1	102.7	85.5
C-GA	1976	19.1	4.2	20.5	105.3	94.5
	1977	19.6	3.6	22.7	109.9	102.0

[a]N X 6.25.

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[Received November 4, 1981]