

FIG. 2. Extraction equipment installation for Southern Soya Inc., Estill, S.C.

FIGURE 2 shows the plant view of the first Stationary Basket Extractor installation at Southern Soya Corporation in Estill, S.C. The extractor with the distillation equipment is shown at the right. The Desolventizer-Toaster is immediately behind the extractor. An external plant view is shown in Figure 3.

The operating results and mechanical performance of the first full plant-operation at Estill have been very satisfactory. The processing advantage of a stationary bed of flakes free from vibration is shown by a clear miscella.

Earlier in this article the French Stationary Basket Extractor was compared to the original vertical

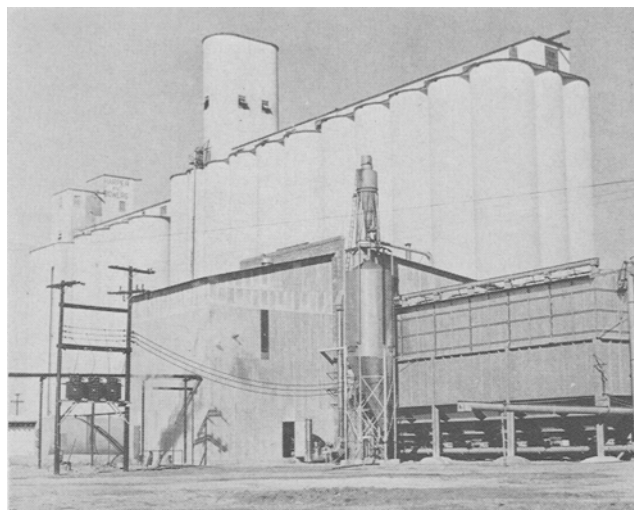


FIG. 3. Plant view, Southern Soya Inc., Estill, S.C.

basket extractors. It is possible to compare it to a still older extraction system, the pot-plant system, which was also developed in Germany in the early 1900's. The pot plant was a series of stationary tanks with screened false bottoms that were charged in turn with solids. Solvent and ever more concentrated miscella were pumped through the pots in series, counter-currently to the charging sequence. Although this system, by modern standards, was costly in labor, it still retains merits of versatility that permit extraction of very difficult materials unsuited to current continuous extractors. The Stationary Basket Extractor can be visualized as a method of combining a group of such extraction pots into a single housing with continuous automatic charging devices for solids and liquids.

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## *Cuphea llavea* Seed Oil, A Good Source of Capric Acid

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FATTY ACIDS in the seed oil of *Cuphea llavea* var. *miniata*, commonly called Cinnabar Cuphea and closely related to the familiar cigar-flower, are reported to be 83% capric acid (1). This shrubby, erect perennial of Loosestrife (Lythraceae) family is native to the foothills and mountains of Sonora and Chihuahua to Oaxaca, Mexico. Because of its showy vermilion flowers it has become a popular ornamental in the southern areas of the United States. Recently preliminary agronomic evaluations indicate

that this species has the potential of becoming an economic source of capric acid; consequently confirmation of the gas chromatographic analysis (1) was sought. Chemical characterization has now established that the major acid is capric. At least 72% of the capric acid present may be isolated in 97% purity by steam distillation of the mixed fatty acids.

#### Experimental

*Extraction of the Oil.* Coarsely ground seeds of *Cuphea llavea* were extracted over-night in a Soxhlet apparatus with 30-60° petroleum ether. The bulk

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of the solvent was evaporated on a steam bath, and the remainder was removed *in vacuo* with a rotating evaporator.

**Isolation of Capric Acid by Steam Distillation.** An 8.79-g. portion of the oil was refluxed with 90 ml. of 0.8*N* ethanolic potassium hydroxide under nitrogen for 3 hrs. Unsaponifiable matter was removed (0.34 g., 3.9%), and free fatty acids were recovered (7.43 g., 84%). The acids were steam-distilled into the following three fractions over a period of 9 hrs.

Fraction	Time (hrs.)	%
Fraction I	(0-1 hrs.)	1.03
Fraction II	(1-5 hrs.)	3.26
Fraction III	(5-9 hrs.)	1.21
Residue		0.75
		6.25 (84% recovery)

Methyl esters from each fraction were prepared by reacting the free acids with diazomethane (2) and were analyzed by gas chromatography (1). The composition of each fraction is listed in Table I.

**Identification of Capric Acid.** The amide was prepared according to the procedure of Shriner, Fuson, and Curtin (3), using 0.331 g. of acid from Fraction II. The yield of crude product was 0.375 g., m.p. 84-94°C. After three recrystallizations from aqueous ethanol 0.110 g. of white crystals, m.p. 96-97°C., were obtained; lit. value 98.5°C. (4). There was no depression of melting point on admixture with authentic capramide.

*Anal.* Calcd. for C<sub>10</sub>H<sub>21</sub>ON: N, 8.2. Found: N, 8.0.

The anilide was prepared essentially by the method of de Jonge *et al.* (5), using a 0.304-g. portion of acid from Fraction II. The crude yield was 0.483 g. of yellow solid. After three recrystallizations from aqueous ethanol 0.256 g. of white crystals, m.p. 62.5-63°C., was obtained; lit. values 69.5-69.9°C. (5) and

TABLE I  
Fatty Acid Composition of *Cuphea havana* Seed Oil and Derived Steam Distillates (Area Percentage of Methyl Ester Peaks)

Acid	Original oil (1)	Steam distillates			
		Fractions			Residue
		I	II	III	
	%	%	%	%	%
Caprylic	0.8	2.4	0.5	.....	0.2
Pelargonic	0.1	0.1	0.1	.....	.....
Capric	82.7	96.8	98.3	93.5	0.9
Unknown	Trace	0.2	0.2	0.1	.....
Lauric	1.2	0.4	0.9	4.7	2.3
Myristic	0.8	.....	.....	1.1	3.8
Palmitic	2.6	.....	.....	9.7	18.8
Stearic	0.5	.....	.....	.....	3.1
Oleic	4.9	.....	.....	.....	.....
Linoleic	6.3	.....	.....	.....	71.0
Linolenic	0.1	.....	.....	.....	.....

62°C. (3). On admixture with authentic capranilide (m.p. 63-64°C.) no depression of melting point was observed.

*Anal.* Calcd. for C<sub>16</sub>H<sub>25</sub>ON: N, 5.7. Found: N, 5.6.

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## Fatty Acids Analysis by High Resolution Nuclear Spin Resonance. A Preliminary Evaluation<sup>1,2</sup>

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High resolution nuclear spin resonance spectra of four fatty acid methyl esters have been presented and discussed from the standpoint of applying this instrumentation to the analysis of the possible eight-component system resulting from the hydrogenation of linolenic acid. If one of the constituents other than 9- or 12-oleic acid can be neglected or found by another method, the analysis seems possible. Experimental conditions for the accurate reproduction of the relative intensities of the spectral bands must first be found.

RECENT ATTEMPTS in these laboratories to hydrogenate selectively linolenic acid in linseed oil to linoleic acid created a need for improved procedures for analyzing fatty acid mixtures (1). This paper is a preliminary evaluation of the usefulness of high resolution nuclear spin resonance (NSR) spectroscopy in the performance of such analyses. Its presentation at this time, in the absence of sufficient data for an analytical paper, is in response to the request by certain workers in the

field of oil chemistry that the results given herein be made more generally available. Spectra from the methyl esters of linolenic, linoleic, oleic, and stearic acids are presented and discussed from an analytical viewpoint. A more general treatment of the application of NSR to oil chemistry has been published elsewhere (2).

#### Presentation and Discussion of the Spectra

The application of high resolution NSR to the analysis of fatty acid mixtures is most simple conceptually as reference to the spectra in Figures 1-4 will show. The spectra may be separated into bands, each of which arises from the hydrogen nuclei or protons in some particular functional group in the molecule. The area under a band, or its intensity, is proportional to the number of protons and hence to the number of functional groups giving rise to the band. Reference to Table I will show that most of the fatty acid methyl esters in the system contribute an unique balance of those functional groups

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