odoriferous constituents in the oil. Failure to remove these constituents completely in the forerun fraction will result in a lower quality product.

Distillation of the main body of the oil was accomplished by controlling the feed line and rotor temps and the oil flow across the rotor. A pot residue fraction of approx 4% was retained by nature of the batch-type equipment. The pot residue could be substantially decreased with large-scale continuous operation equipment. Typical batch distillation data on the oil processed in this manner are given in Table II.

Analysis of the distilled oil indicated that there was no destruction of the heat-labile constituents. Free fatty acids and odor constituents of low mol wt were removed in the forerun fraction, and high boiling polymeric substances and the bulk of the pigments were retained in the pot residue. The main fraction was lighter in color and almost free of odor as compared to the undistilled oil. Visible spectra of the winterized oil and molecularly distilled oil are shown in Figure 1.

Combined Bleaching and Distillation

Fish oil that has undergone adsorptive bleaching followed by molecular distillation would be expected to be superior in color, odor, free fatty acid content, and polymeric substances to fish oil that had been processed by only one of these techniques. Experiments were designed to give insight into the nature of such a dual processed fish oil.

Winterized menhaden oil was bleached with the Canadian earth (cf. Table I) at 80C for 30 min, as established in the preliminary experiments. Quantities of oil up to 7 gallons were treated in a 10-gallon stainless steel reactor equipped with stainless steel stirrer. The vacuum of the system was controlled at 100 mm Hg, using a mechanical pump and a suitable moisture trap. An 8% clay concn was used, because it was found to produce a high quality oil product from subsequent molecular distillation.

Figure 1 shows the visible spectrum of winterized menhaden oil bleached with 8% clay, followed by centrifugal molecular distillation of a 5-gallon batch. The product of this treatment is pale yellow, nearly odorless, and very bland in taste. It was found that the odor-flavor character of the treated oil was dependent on how carefully the forerun fraction was removed. The color is influenced by the final temp of the distillation. Attempts to force high distillation rates with unnecessarily high temp result in distillation of yellow-colored substances.

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Processing Ironweed *(Vernonia anthelmintica)* Seed in a Soybean Extraction Pilot Plant[1]

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Abstract

High quality domestic *Vernonia anthelmintica* (ironweed) seed was grown on many experimental test plots at a variety of locations during the 1963 season. Quality appeared to be related to seed density as judged by high oil content of 25 to 32%, the oils having oxirane oxygen values of 3.6 to 4.0% with low acidity, less than 2% calculated as epoxyoleic acid. Larger plantings in 1963 made without benefit of test plot data produced poor quality seed. For processing some of this seed was upgraded to fair quality by air-elutriation. Processing in a small soybean continuous solvent extraction plant was successfully achieved with only minor changes in existing equipment. No enzymatic lipolysis occurred during these operations. The oil obtained was equal in quality to that prepared from the same seed in the laboratory by the best procedures available. Also, the oil was improved in quality by removal of the major portion of its free fatty acids and unsaponifiable material. The chief natural component of the seed oil, trivernolin, was prepared from a substantial quantity of the oil miscella to demonstrate the commercial feasibility of this operation.

Introduction

NEW CROPS RESEARCH at the Eastern Regional Research Laboratory has been directed towards development studies on epoxy-bearing seed oils. A series of reports from this laboratory has appeared related to work on imported *V. anthelmintica* whose seed has held from 18 to 27% oil; about 65–70% of the oil has been composed of the single compound trivernolin, the triglyceride of vernolic (12,13-epoxyoleic) acid. The reports mentioned have reviewed the literature on *V. anthelmintica* (1); described procedures used for the production and purification of the oxygenated fatty components, trivernolin, 1, 3-divernolin, vernolic acid, methyl vernolate and 12, 13-dihydroxyoleic acid (2–5); presented information on a satisfactory gas-liquid chromatographic column for the determination of epoxyoleic acid in seed oils (6), and given accounts of evaluation studies on the stabilizing properties of some *Vernonia* products (7, 8).

[1]Presented at the AOCS Meeting, Chicago, October 1964.
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TABLE I

Results of *Vernonia* Seed Fractionation by Laboratory Air Separator

Sample	Approx quantity	Oil in seeds	Oil	
			Oxirane oxygen	FFA
	g.	%	%	%
Original A	100	20.5	3.44	11.6
Light fraction	33	12.1	2.80	25.2
Medium fraction	11	18.7	2.97	19.5
Heavy fraction	56	28.0	3.63	6.6
Original B	100	19.6	2.46	25.8
Light fraction	15	2.9	1.58	36.8
Medium fraction	60	15.0	2.49	27.7
Heavy fraction	25	28.0	3.29	16.3

The objective of this report is to describe the initial processing of domestic *V. anthelmintica* seed in a small soybean plant. The report also includes an account of the refining of seed oil to upgrade it for evaluation studies and the preparation of trivernolin from an aliquot of seed oil miscella to demonstrate the commercial feasibility of its production, a process previously described (1,5).

Upgrading Vernonia Seed for Processing

During the analysis of samples of *Vernonia* seed grown in test plantings throughout the country in the 1963 season, it was noted that there was a correlation between apparent seed density and quality, with the more dense seeds being higher in oil and oxirane oxygen content and lower in free fatty acids. Attempts were made to fractionate seeds on the density basis first by floatation in water, then by air elutriation. A seed sample which contained 23.7% oil, 3.38% oxirane oxygen and 8.55% free fatty acids (FFA) (calculated as vernolic acid) was placed in water and those seeds which floated were separated by decantation. The two fractions were dried and analyzed with the following results:

	Oil	Oil	
	Oil	Oxirane oxygen	FFA
	%	%	%
Light fraction	19.8	3.08	12.63
Heavy fraction	31.3	3.69	1.82

This showed that seed lots could be upgraded materially by removing lightweight, immature seeds. Because separation by floatation in water was not practical for large lots and because it did not provide any latitude in the fractionation process, a small laboratory air fractionating column was constructed from a piece of glass pipe 2 in. in diam and 4 ft long.

One hundred-gram samples of two bulk shipments of seed A and B were placed in the laboratory air fractionator and separated into three fractions by gradually increasing the air velocity through the vertical glass pipe. The data for the original seed samples and three fractions are shown in Table I.

The goal was to obtain material for the commercial seed processing test which contained more than 25% oil with over 3.6% oxirane oxygen and less than 2% free fatty acids. The heavy fraction from sample A, which accounted for a little over 50% of the whole, had satisfactory oil and oxirane contents but was higher than desirable in free fatty acids. Sample B failed to yield a fraction suitable in either oxirane or acid content. It should be noted that these seed samples were lower in quality than those grown in some areas of the country, notably Georgia and Kentucky, as shown in the recent report by White and Haun (9) on the results from the 1963 test plantings.

TABLE II

Fractionation of *Vernonia* Seed in Commercial Seed Cleaning Equipment

Sample	Approx quantity	Approx density lb/bu	Oil	Oil	
				Oxirane oxygen	FFA
	lb		%	%	%
Original A	1500	30	20.5	3.44	11.64
Light fraction-1	660	22	9.9	2.81	20.72
Heavy fraction-2	840	35	25.3	3.49	8.89
Light-2a	250	30	18.4	3.04	16.8
Heavy-2b	590	39	26.6	3.59	5.34
Original B	580	27	23.7	3.38	8.55
Light fraction	330	20	9.5	2.67	14.19
Heavy fraction	250	39	38.7	3.57	4.35

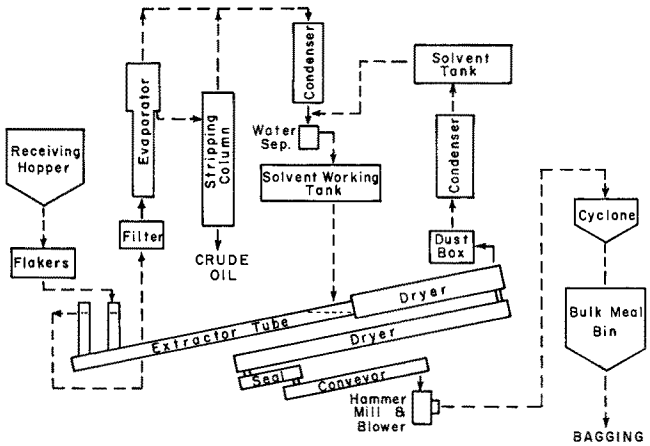

FIG. 1. Continuous solvent extraction of *Vernonia* seed.

Based on the results of tests with the laboratory air fractionator, the Philadelphia Seed Company, commercial seed cleaners, was asked to attempt to separate two bulk lots of approx 1500 to 580 lb into light and heavy fractions in the hope that the heavy fractions would be of good enough quality to use in a commercial plant extraction test run.

Results of the fractionation in the commercial seed cleaning equipment are shown in Table II. Because the first heavy fraction from sample A was too high in free fatty acids, it was subjected to a second fractionation making approx a 2 to 5 split. As the results in Table II show there was some improvement in the quality of the second heavy fraction but the acid content of the oil was still high.

Equipment Used in Seed Processing

The equipment used was originally designed to process soybeans by continuous solvent extraction at the rate of 400 lb/hr. Due to economic conditions, the plant is now used for specialty extraction work such as the processing of wheat germ, sunflower seed, and pilot plant work.

The equipment consisted of a conventional Ford-type extractor enclosing a ribbon conveyor with total immersion continuous solvent (n-hexane) extraction. Tempering before flaking was not required for *Vernonia* seed processing nor was heat used during the extraction (5). The conveyor was provided with two vertical standpipes at the lower end as indicated diagrammatically in Figure 1; one of these was used for the admission of fresh flakes and the other for miscella overflow.

The entire plant was semiautomatically controlled and prior to its use for processing *Vernonia* seed had been thoroughly cleaned so that only a minimum amt of contamination would be encountered from its previous use in the extraction of wheat germ. The forerun and tail fractions whose weights appear in Table III were not considered typical; hence, analytical data obtained on these fractions have been omitted from Table IV.

TABLE III

Processing *Vernonia* Seed for Oil
Material Balance

		Quantity	
		lb	lb (mfb)
Gross seed	(IN)	759	717
Meal	(OUT)	487	469
Fines	(OUT)	60	56
Crude oil forerun	(OUT)		21.7
Main fraction	(OUT)		149
Tail fraction	(OUT)		18.2
Total			713.9 (0.4% loss)

Oil yield		Meal analyses		
	%		Oil in %	Protein in %
From seed processing	26.3			
By analysis of seed	27.2	Meal	2.2	12.5
Recovery	96.7	Fines	1.3	56.3
Oil loss in processing (by difference)	0.9			
Oil loss (calc'd. from analysis of meal and fines)	0.3			

Results and Discussion

A. *Vernonia Oil Production and Refining.* The *Vernonia* seed used in processing consisted of a mixture of 559 lb designated as "A-2b" and 200 lb of "B, heavy fraction" in Table II, a total of 717 lb on a moisture-free basis.

Analytical values for the mixture of seed processed were: 27.2% oil (dry basis), 3.58% oxirane oxygen for the oil, and 5.07% FFA in the oil.

Two processes have been offered recently (5) as a means of controlling lipolysis during *V. anthelmintica* seed extraction, one a rapid n-hexane extraction at room temp of freshly flaked seed, the other a heat inactivation treatment preferably by autoclaving prior to flaking. The former method was used in this processing test being favored by winter weather conditions. The seed fed to the hopper was at 36F, the temp maintained in the plant during extraction. The solvent temp increased to 60F at the end of the extraction. The seed was fed at a slower than normal rate, 100 instead of 400 lb/hr to handle the small batch efficiently. About 1 hr was required for seed to pass through the processing operation ending up as oil and dried, bagged meal (Fig. 1). On-the-spot analyses of miscella at intervals during processing showed the FFA content to be 5.04–5.05% in close agreement with one of 5.07% obtained by previous laboratory analysis of oil extracted from the seed.

The material balance in the processing of *V. anthelmintica* seed for oil is presented in Table III. The main oil fraction containing 149 lb of oil was retained in the form of a miscella in about 50 gal of solvent. An aliquot containing 42.5 lb of oil was removed for use in the preparation of trivernolin. The remainder of the main fraction miscella containing 106.5 lb of oil was diluted to 55 gal with n-hexane to eliminate air space. Refinement of this fraction was carried out at the Eastern Regional Research Laboratory. The procedure used in this refinement consisted of (*a*) the removal of the major portion of the unsaponifiable material following a procedure previously described (5), (*b*) the removal of FFA with dilute methanol solution of potassium carbonate, and (*c*) treatment with an adsorbent mixture to remove color and odor.

Briefly described this refinement was as follows: The *Vernonia* miscella containing 106.5 lb of oil was diluted with n-hexane to about 70 gal upon arrival at the Eastern Regional Research Laboratory. The mixed glycerides which precipitated out of solution at −76F with continuous stirring during overnight storage were filtered off and washed three times with cold solvent at −76F. The unsaponifiable material remained in the filtrate and washings; these were combined for solvent removal. Freed of solvent, this material weighed 8.1 lb (oxirane oxygen 0.14%; FFA 11.3%, iodine value 226.2); its chemical composition is under investigation. This large amt of unsaponifiable material is in agreement with the quantities previously reported (1,5). To remove FFA from the mixed glyceride fraction the wet filter-cake was dissolved in 21 gal of n-hexane at room temp. This solution was washed three times with a solution containing 1.3 lb of potassium carbonate in 2 gal of water and 1 gal of methanol. No emulsion problems were encountered. The n-hexane layer was separated and treated with a mixture containing 10 lb of Filtrol No. 4 and 5 lb of Darco G-60 and held at about 105F with stirring for 10 min. The adsorbent mixture was filtered off on Republic Seitz No. K-5 filter pads through a filter press under pressure. The solvent was removed from the filtrate under vacuum after drying the solution over anhydrous sodium sulfate; yield was 75.3 lb of refined oil with analyses shown in Table IV. The data on the refined oil fraction compare favorably with those previously published (5).

TABLE IV

Vernonia Oil Analyses

	Main fraction	
	Crude	Refined
Oxirane oxygen (%)	3.56	4.09
FFA (%)	5.5	0.20
Iodine value (Wijs)	106.7	91.7

B. *Preparation of Trivernolin.* The 42.5 lb *Vernonia* oil aliquot reserved for trivernolin production was diluted to about 30 gal with n-hexane. This solution was stirred with 4 lb of Filtrol and 2 lb of Darco for 15 min and then filtered through a Sparkler filter press using K-5 filter pads. The filtrate was held at 10F overnight with constant stirring. The trivernolin which crystallized during this period was strained off at 10F through a canvas filter and returned to the Eastern Regional Research Laboratory for further refinement. This was achieved by low temp crystallizations from n-hexane in a manner similar to that previously described (1,5) with comparable yield and purity.

Some of the refined *V. anthelmintica* seed oil and the trivernolin have been distributed for commercial evaluation in plastic formulations. Quantities of both products have been reserved for the preparation of derivatives and evaluation studies in progress at this laboratory.

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