gies of both $\beta'$ states appear to be equal. Whether the 2 $\beta$ phases possess nearly identical solid-state structures at the crossover temperature cannot be determined from our data. Such a conclusion requires determination of molecular volumes, coefficients of expansion, and the energy of the $\beta_2'$ to $\beta_1'$ phase transition.

These results on individual solid phases of monoacid triglycerides reinforce evidence that even and odd chain-length saturated triglycerides exhibit distinctly different polymorphic behavior. An explanation of the $\beta'$ specific heat anomaly observed in this work might also identify reasons for the alternation of physical properties between the $\beta'$ states of even- and odd-membered triglycerides.

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# ☙Separation of Seed By-products by an AC Electric Field

**LOUIS C. WEISS** and **DEVRON P. THIBODEAUX**, Southern Regional Research Center, USDA, New Orleans, LA 70124

## ABSTRACT

A method for separating constituents of the by-products derived from agricultural processes offers the possibility of recovering their valuable components. Partial sorting by a unique electric field enriches the protein concentration in sunflower-seed meal and cottonseed meal by removing objectionable components such as hulls or gossypol. Enrichment is accomplished by the dynamic forces from an electric field that is created by a set of parallel electrodes encased in a plastic panel and connected to an AC power supply. When the panel is activated, particles on its surface become charged, levitated and transported. When a mixture of particles contacts the panel, a difference in the charge is imparted to the components, opposite directions of transportation occur, and, then, the particles separate. The separation, or sorting, offers an additional possibility for upgrading agricultural products. Properties measured for these products and their separated components are charge, charge-to-mass ratio, size, size distribution, shape, moisture, nitrogen and gossypol. This new and direct method of separating components seems independent of their size and shape, yet it is probably dependent on the charge-to-mass ratio. The single-stage separation is an analytical tool that might become an engineering process. The electric panel of the contact type contains, transports and refines materials—all without a cup, conveyor or filter touching the sample. Its promise and versatility warrant considering the panel by itself and in conjunction with other processes.

## INTRODUCTION

In our research to apply electric fields for the processing and evaluation of textiles, a significant accomplishment was the induction of electrical charges in cotton fibers (1). Using the concept that electric fields act without the need of a mechanical continuum, a method was developed to map the contours of such "wrinkled" surfaces as durable-press fabrics (2). Most recently, the traveling-wave, electric-curtain concept, as developed by Masuda (3), was successfully applied to the control and movement of different fibers (4). The forces of an electric field can reduce the energy consumed in moving machine parts, conserve the space required in aerodynamical processes and isolate work areas.

While considering problems associated with the transport of materials by a traveling-wave, a previously unreported effect was discovered. The observation was made that most materials of a mixture, when placed in an electric curtain, move in one direction; yet, a small fraction, acted on by a reverse force, moves in the opposite direction, resulting in separation or sorting. Described here are such significant separations as extracting gossypol from cottonseed flour, minerals and pollen from the dust of mills and hull waste from sunflower-seed powder. One goal of this report is to demonstrate that potentially useful separations can be accomplished solely with an AC electric field. A second goal is to use the experimental evidence to develop an understanding of this phenomenon and why it occurs.

## MATERIALS AND METHODS

### Samples

Three different agricultural mixtures, by-products of processes, were used to determine the degree to which an electric curtain could separate components. One material chosen was crushed sunflower seed, the by-product of a commercial oil-extraction process. The crushed seed consists of potentially valuable seed proteins mixed with fragments of the seed hulls. The similarities in the size of these components render separation by normal procedures difficult. The second sample was the waste or unders from the liquid cyclone process, which produces protein enriched flour from cottonseed. These unders are essentially a mixture of cottonseed proteins, pigmented particles of almost pure gossypol and some protein particles contaminated with gossypol. The third sample, waste collected at the baghouse of a commercial grain elevator, was a multicomponent mixture of grain, dust, hull and chaff from wheat.

### Procedures

Ca. 4 g of material were fed onto the surface of the panel. Separation was accomplished by first subjecting the sample to a relatively small charging voltage for ca. 40 sec then increasing the voltage by $\frac{1}{3}$ for ca. 10 sec. Voltage was then reduced to the original value. This impulse-cycle technique was repeated until separation was obtained. The voltage value depends on the materials to be separated and the environmental conditions. Sunflower seed and wheat grain usually require a charging voltage of 4 kV, with a slightly higher voltage for cottonseed.

Experiments were conducted at ambient conditions of ca. 21 C and a relative humidity of less than 55%. After each experimental run, the separated fractions were collected, weighed and subjected to the following analyses: mass-separation efficiency, charge-to-mass ratio, nitrogen content, moisture regained, particle area, circularity index and gossypol content.

Separation efficiency, determined by collecting and weighing the 2 separated components, is calculated as a fraction of the starting material (referred to as the parent). Charge-to-mass ratio is based on a specific charge determined by collecting the selected fraction in a "Faraday Cup" placed at the edge of the plate and grounded through an Electrometer (Keithley 610B). The material collected in the cup is weighed and the charge per unit mass is determined.

The nitrogen content, determined by the Kjeldahl method, gives an index of the protein content of the separated components and their parent. The moisture regained or water absorbing ability of each sample and the gossypol content of the cottonseed were determined by standard methods (5,6). Each of these properties is expressed as a fraction of the sample's oven-dry weight.

The size and shape parameters of parents and separated components were obtained using an Image Analyzer (Quantimet 723). This provides a statistical analysis that computes the mean value for the projected, cross-sectional area and standard deviation for each sample. With this area, A, the analyzer also measures a perimeter, p. As the ratio $4\pi A/p^2$ approaches 1, the shape of the area approaches a circle (7).

### Process

In the Masuda-type traveling-wave apparatus, parallel sets of individual conductors connected to an AC electrical source form an undulating electric field in the surrounding air. This series of nonuniform electric waves is named the "electric curtain." If particles are charged and then placed in this curtain, they are confined without touching a barrier. But, if the particles are poorly charged, some loss occurs. Placing a dielectric sheet over the conductors not only prevents these losses but automatically causes an interchange of electrons among the particles and perhaps with the sheet. A significant electrical charge is established on these particles, making external charging unnecessary. If 3 phases of an AC voltage are connected successively to the different conductors, a series of traveling electric-waves are created. Material within the field is carried along as if on an invisible conveyor. An apparatus like the electric grid covered with a dielectric sheet is called a "contact-type, electric-curtain panel" or, more simply, an electric panel (4,8). In this investigation, nonconducting materials are simultaneously charged, levitated against gravity and transported by the electric panel.

A schematic diagram of the circuit and panel is presented in Figure 1. In the B section, the solid lines represent 3 sets of conducting grids and the dashed line indicates the dielectric sheet or excitor that encapsulates all conductors.

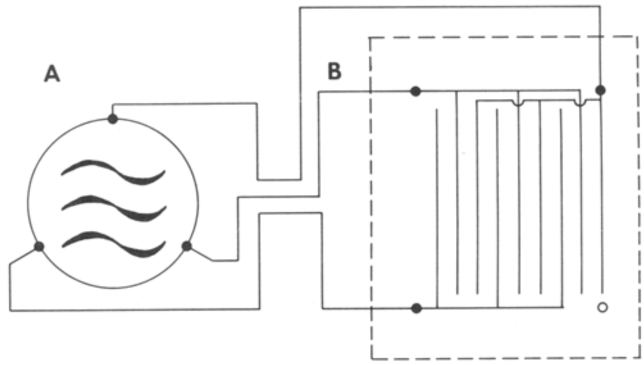


**FIG. 1. Simplified schematic of power and panel—A represents the electrical source and B illustrates the wiring in an electric panel.**

The panel is 0.09 $m^2$ or a square foot in area with a 3 mm space between grid conductors. The power source, A, is a 60 Hz, 3-phase, high voltage supply that varies continuously between 0-30 kV. The present range is only the lower third of the design. Details of the electrical circuit, its operation and panel construction have been published (4).

### Theory

What are these forces that move a body without mechanical or aerodynamical means? They are caused by the electric field and, in theory, can be represented by the following equations:

$$m\frac{d^2x}{dt^2} + D\frac{dx}{dt} = q\,E_x \,\mathrm{Cos}(2\pi ft) \qquad [1]$$

$$m\frac{d^2y}{dt^2} + D\frac{dy}{dt} = q\,E_y \,\mathrm{Cos}(2\pi ft) + mg \qquad [2]$$

Briefly, a body is charged by an interchange of electrons and undergoes the motion expressed on the left-hand side of each equation while under the influence of the driving forces on the right-hand side. With this notation, when the equations are integrated, the concept of phase angle is introduced. In an x-y plane, where z is equal to a constant; a mass, *m*, undergoes an acceleration simultaneously with a dissipative force that acts according to the mass' velocity. This dissipative force is also a function of the drag constant, *D*, which, in turn, is proportional to the radius of a particle. The driving force is the charge, *q*, times the x-component of the electric field, where *f* is the frequency and *t* the time. The second equation is analogous to the first and has an additional term, *mg*, which is the accelerating downward force caused by gravity, *g*. A lighter mass, a greater charge or an increase in field strength will increase the motion a body displays in the field.

Numerical analysis offers a solution for the motion of the body, *m*. Assume a small displacement for x and y, calculate the results, store and repeat the calculations. A plot of these results in Figure 2 shows such a path of motion for an ideal point of mass as it oscillates about its equilibrium at a height of 8 mm above the panel. The grid conductors are shown as dots in the cross-section on the x-axis. The magnified inset shows a particle undergoing a rotational oscillation in the x-y plane according to the preceding set of equations. The net force of repulsion produces the levitation effect. Here, the rotation is in a clockwise direction with a transverse-wave motion toward the y-axis. The velocity of rotation is obviously greater than the velocity of translation. Such a translation-rotational path closely approximates a prolate cycloid. The length of panel covered during 1 cycle, which could be called a wavelength, is ca. 11 mm, with the velocity of a particle 0.68 m/s.

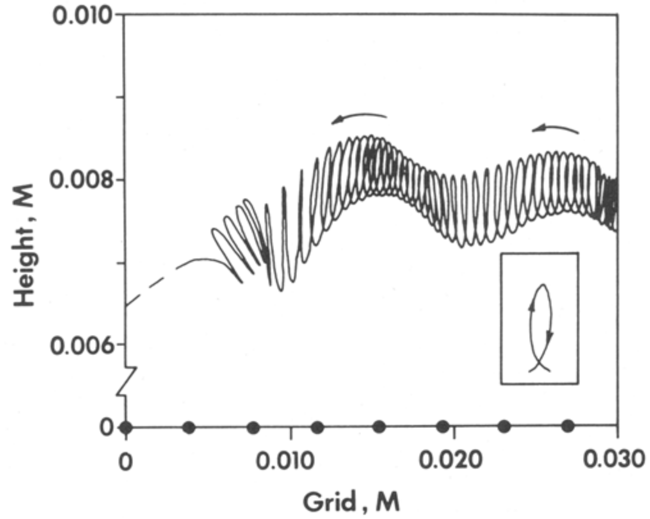


FIG. 2. Computer simulation for the path of a center of mass tracing height as a function of distance along the panel.

### Separation

Theory predicts motion in only one direction. However, under the conditions previously described, some materials nearest the surface of the panel were transported in the opposite direction. If the panel was considered to be a linear electric motor, then this reverse force would be analogous to a "back emf" or counterproductive, electromotive force. Reasoning that this backward force should be greater for a mixture of powders, mixtures that often occur as the by-products of our agricultural system were tested for separation. For the first time, significant separations or refinements were achieved solely by an electric panel. Notable among these were the extractions of pigmented gossypol from cottonseed flour and of hulls from sunflower-seed powder.

In Figure 3, the electric panel is represented by the letter C with the location where particles are fed onto the panel indicated by the area D. Directly above and along the length of D is a representation approximating the motion of a cloud of particles. When the panel is activated in the separation mode, the majority of a sample is transported in the expected direction, A. Using a water-wave analogy, this is defined as the surfing direction. A smaller amount—usually a more homogenous component—is transported in the opposite direction, B. This amount is said to be caught in the undertow. In one of his theories, Masuda stated that separate movements with different velocities might be found, but, they were thought to be in the same direction (3). Speculation of opposing velocities, such as observed here are not found in the literature.

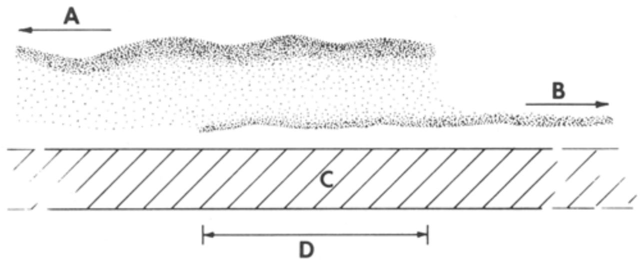


FIG. 3. Representation of the paths for separation of a bicomponent mixture—A indicates surfing direction and B the undertow; C represents the electric panel, which receives the sample in the region above D.

## RESULTS AND DISCUSSION

One by one, wastes from agricultural processes were fed onto the panel. Components were separated, collected and their specific charge and percentage of nitrogen determined. Results for these samples are presented in the first columns of Table I. Their dimensional values of size and shape are listed in the last columns. Section A of the table presents the values for lycopodium, a reference material that approaches the ideal particle for gauging electrical activity. Lycopodium is a naturally occurring dielectric with a nominal diameter of 30 μm and serves to standardize panel performance. When the electric panel is operated at 5 kV, a charge of about $10^{-16}$ coulombs per lycopodium particle is determined. During this time, the particles couple well with the traveling wave and move in the surfing direction. Of all the particles tested, those of the positively charged lycopodium are the smallest, the most uniform and the most spherical.

**TABLE I**

**Some Properties of Selected Seed By-Products**

| Component | Charge/mass (nc/g)[a] | Nitrogen × $10^2$ (gN/g) | Moisture regained (%) | Projected area | | |
|---|---|---|---|---|---|---|
| | | | | Mean ($mm^2$) | SD ($mm^2$) | Circularity ratio |
| A. Lycopodium[b] | | | | | | |
| (pure) | +500 | 1.33 | 3.25 | $0.71 \times 10^{-3}$ | $0.12 \times 10^{-3}$ | 0.90 |
| B. Sunflower seed[c] | | | | | | |
| Parent | – | 4.70 | 8.67 | 0.269 | 0.264 | 0.78 |
| Meal | +175 | 5.19 | 8.60 | 0.349 | 0.451 | 0.74 |
| Hull | -3,000 | 3.24 | 8.78 | 0.255 | 0.199 | 0.75 |
| C. Cottonseed[d] | | | | | | |
| Parent | – | 8.50 | 8.52 | $0.359 \times 10^{-2}$ | $0.668 \times 10^{-2}$ | 0.79 |
| Meal | +1,420 | 9.70 | 9.79 | $0.223 \times 10^{-2}$ | $0.580 \times 10^{-2}$ | 0.81 |
| Gossypol | -10,124 | 8.08 | 8.00 | $1.067 \times 10^{-2}$ | $1.282 \times 10^{-2}$ | 0.78 |
| D. Grain dust[e] | | | | | | |
| Parent | – | 2.12 | 9.34 | 0.352 | 1.080 | 0.74 |
| Conglomerate | +540 | 2.01 | 9.26 | 0.461 | 0.808 | 0.70 |
| Hull | -720 | 2.00 | 10.38 | 0.309 | 0.313 | 0.72 |
| Wheat | +0.04 | 2.17 | 9.02 | 4.803 | 3.14 | 0.83 |

[a]nc is nano (or $10^{-9}$) coulombs.
[b]Standardizes electrical activity.
[c]Parent is by-product after solvent extraction.
[d]Parent is unders from liquid cyclone.
[e]Parent is waste from elevator baghouse.

### Sunflower Seed

After solvent extraction of the sunflower-seed oil, a powder of crushed components accumulates. Data for this by-product of sunflower seed are presented in section B of Table I. When the parent is placed on the electric panel, it is charged, levitated and transported. Simultaneously, separation into 2 components occurs. The larger volume is a component of refined meal moving in the surfing direction. Another component, which is smaller in volume, moves in the opposite, or undertow, direction. It is composed essentially of hull fragments and is the least variable in appearance. The hull component greatly differs in appearance from the meal component or their parent mixture. The separation is not absolute but is rather a selective concentration of components at opposite ends of the panel. Sunflower-seed powder is among the most active material tried on the panels to date. The meal component acquires a net positive charge, whereas the hull component acquires a negative charge. The meal-enriched component has an increase in nitrogen, indicating an increase in its concentration of protein. Correspondingly, nitrogen concentration in the hull component decreases. This single-stage process yields a meal component with 0.02 g nitrogen/g wt more than is found in the separated hulls, an increase of 60%. Nitrogen rankings for the parent and both components of this seed display promising results.

Data are presented in the table for the size and shape of these sunflower-seed components. The mean values of the areas for these 3 specimens imply that hulls are the smallest and the standard deviations (SD) indicate that hulls are also the least variable. The SD show that the distributions in sizes are much greater than the differences among the means, or, that the components do not significantly differ in size. The area-to-perimeter ratios indicate that all samples are fairly circular, with the meal and hull components similar in shape. Image analysis allows one to infer that neither size nor shape play a major role in the separation of sunflower seed achieved by this electric panel.

### Cotton Seed

When the unders from a liquid cyclone process are fed onto the panel (9), the component that is lighter in color is transported in the surfing direction. In contrast, a second component moves in the opposite direction. These latter particles are mostly black, pigmented gossypol. The results for cottonseed, presented in section C of Table I are similar to those for sunflower seed. The charge per mass for the transported meal is positive. For the separated component, gossypol, the charge per mass is negative. Again, an increase in nitrogen per unit weight for the meal over its parent and over the gossypol-enriched component is found. The meal component has 0.016 g of nitrogen more than the gossypol component for each gram of sample weight. This 20% improvement in nitrogen concentration is similar to but less than that achieved with sunflower seed. The percentages by weight for gossypol were also determined for the cottonseed samples. The parent contained 16.2 mg of total gossypol per gram of sample. The electric panel increased this fraction by 18% for the gossypol component and reduced the meal component by 32%. Measurements for free gossypol followed the same trend.

When comparing the mean areas for the cottonseed unders, the meal component has the smallest size and the gossypol component has the largest size. These measurements reflect previously reported qualitative observations on protein and gossypol (10). Yet, the SD show that the components do not significantly differ in size. The circularity ratios indicate that all samples have about the same shape. Again, image analyses imply that neither size nor shape plays a major role in this separation.

### Wheat Grain and Other Materials

The wheat grain was obtained as dust from the baghouse of a commercial grain elevator. The panel unexpectedly separated this grain dust into 3 components—hulls, which were ejected into the undertow; fragments of grain, which remained on the panel; and a conglomerate, which rose and surfed. The latter was composed of grain, dust and chaff (parts of the light, exterior shell). Section D of Table I presents a summary of results for the waste from wheat grain. The hull component was negatively charged and the conglomerate was positively charged. The negligibly charged wheat fragments that did not levitate possess the highest nitrogen, the smallest charge-to-mass ratio, and the largest size. These fragments, which are in a class by themselves, could be removed by a sieve. The lack of an increase in nitrogen for the conglomerate is probably because the conglomerate is a mixture. Later, we observed that a fourth component, chaff, could be removed by an air flow perpendicular to the conglomerate's direction of flow.

Image analyses indicate that the hull component has the smallest average size and highest uniformity. The parent waste is the most variable in size, reflecting the fact that it consists of at least 4 components. The conglomerate is next in variability, probably because of its chaff. For this waste and its 2 components, the differences among the mean areas are much less than the ranges indicated by their standard deviations. All 3 approach a circular cross-section at about the same degree. Neither size nor shape significantly affects this electrical separation. Most important is the fact that the electric panel alone separates this waste into 3 components. Also, when an aerodynamical force was combined with the panel, the fourth component was resolved.

Three additional by-products were subjected to the electric panel. First, in mixture of cottonseed flour contaminated with *Aspergillus niger,* the flour containing this black mold was vigorously ejected. A more difficult task was that of separating cotton linters from a mixture with small shreds of plastic. The last separation was of cotton trash collected from beneath the card at a textile mill. The vigor of this separation varied for different particles. When separated, each component carried some respirable dust.

### Comparisons

Classifying observations resulting from this new process should aid our understanding of it and lead to future applications. Seed components that are smaller in size are more uniform, components that are larger are generally less uniform. The improved or refined meals from both cottonseed and sunflower seed more closely resemble their parents in appearance than do the hulls or gossypol. The meals acquire the same sign or polarization of charges. The charge-per-mass ratio for the meal components of both seeds and the grain are positive, whereas the fragments of hulls from sunflower seed and grain, along with the gossypol from cottonseed, are negative. The resulting increase in nitrogen implies an increase in the protein content for each of the 2 meal components and the hulls and gossypol that are ejected show a decrease in nitrogen concentration. When the parent for sunflower seed or cottonseed is completely separated, a weight loss of ca. 7% occurs. Using this, and multiplying the components' weights by their concentrations, shows that the total nitrogen is conserved during these separations.

The dual action of increasing protein and removing objectionables offers potential uses of this process in upgrading sunflower-seed powder to an animal feed, reclaiming meal from grain waste and, perhaps, upgrading cottonseed from a feed to a food. As the panel also increases the concentration of gossypol from cottonseed unders, the process may be suitable as an improved source of gossypol, which is becoming useful in several new areas of research.

Water content, often important in mechanical processing, is also an important factor in electrical processing. The moisture regained in these samples after drying indicates that hulls from sunflower seed and grain have the greatest water-absorbing ability. Agreeing with the known deleterious effect of moisture on electric fields, their translational speeds during separation seem to be the slowest. In contrast, the low moisture regain of the reference lycopodium is reflected in its vigorous action over the panel. The effect of moisture is opposite to and probably less than that of the charge-to-mass ratio. The reduced vigor of pigmented gossypol, however, must be attributed to another property, perhaps its different dielectric characteristics or the high negative value of its specific charge.

### Interpretations

As to how and why separation occurs, consider some details of this process. The undulating traveling wave is composed of electric field lines emanating from each conductor of the grid. These lines of potential are stronger nearer the panel than they are at a distance. The electric forces grow from a complex, near-field mode to a simpler, far-field mode. In the process of separating particles, each component comes to equilibrium against gravity at a different mean height above the panel, depending primarily on its charge-to-mass ratio. All components enriched in protein rise to a greater height than the ejected components. The protein components couple with the far-field mode of the wave and ride in the surfing direction. Sunflower seed and wheat hulls, along with the component enriched in pigmented gossypol, reach equilibrium near to and sometimes on the panel. These particles couple with the more complex mode of the wave, are caught in the undertow and move in the opposite direction. Hence, particles are equilibrated according to the properties that affect their charge-to-mass ratio. The particles then couple with the mode of the traveling wave that dominates at that height and are moved accordingly.

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# Ximenynic Acid in *Santalum obtusifolium* Seed Oil

**J.R. VICKERY, F.B. WHITFIELD, G.L. FORD** and **B.H. KENNETT**, CSIRO Division of Food Research, P.O. Box 52, North Ryde, N.S.W. 2113, Australia

## ABSTRACT

Analyzing *Santalum obtusifolium* seed oil led to the identification of 12 fatty acids; only ximenynic (71.5%) and oleic (14.3%) acids were present in substantial amounts. Data on the mass spectrum and $^{1}H$- and $^{13}C$-NMR (nuclear magnetic resonance) spectra of methyl ximenynate are given.

## INTRODUCTION

*Santalum obtusifolium* R. Br. (family Santalaceae) is an erect glaucous shrub 1-2 m high that grows in the Hawkesbury sandstone region of eastern New South Wales (N.S.W.), Australia. The fruits are drupaceous and the average mass of the dry seeds is ca. 200 mg.

Ximenynic (santalbic) acid, *trans*-11-octadecen-9-ynoic acid, is an important constituent of the seed oils of *Ximenia, Santalum* and *Exocarpos* genera of Santalaceae and has been described by Ligthelm et al. (1,2), Gunstone and Russell (3) and Hatt and Szumer (4).

The purpose of the study was to determine whether this acid occurred in the seed oil of *S. obtusifolium* and to substantiate chemical studies with data on its nuclear magnetic resonance (NMR) spectra.

## EXPERIMENTAL PROCEDURES

### Material

Seeds were collected by L. V. Langley, Robertson, N.S.W.

### General Procedures

The methods used for extracting the oil, preparing methyl esters, the Halphen color test and gas chromatography (GC) were similar to those described in 2 previous papers (5).

The methyl esters were hydrogenated by dissolving 5 mg in 5 mL hexane, adding Adams' catalyst and bubbling hydrogen through the solution for 15 min at 20 C.

### Separation and Identification of Methyl Ximenynate

The methyl ester of the fatty acid was isolated by argentation thin layer chromatography (TLC) at −20 C, using 90/10 v/v toluene-hexane as the developing solvent. The ester, 95% purity by gas liquid chromatography (GLC), appeared under short-wave ultraviolet (UV) light as a medium-dark band near the top of the plate before the dichlorofluorescein spray solution was applied. Samples of the ester for spectroscopic examination were further purified by GLC by methods previously described by Whitfield et al. (6), using a stainless-steel capillary column (150 m long, 0.75 mm i.d.); the walls coated with silicone OV-101. The UV spectrum of the ester (in hexane) was recorded on a Gilford 2600 spectrophotometer, the infrared (IR) spectrum (as a thin film) on a Perkin Elmer 521 spectrometer and the $^{1}H$- and $^{13}C$-NMR spectra (as $C_6D_6$ solutions) on a Bruker CXP100 spectrometer. The low-resolution and high-resolution mass spectra were recorded using a Varian MAT-311A mass spectrometer. The products obtained from the