The importance of solubilization is best assessed by comparing data for the same solubilizate involved in the soil removal systems. Such data are given in Table V for tristearin solubilization and removal from the frosted glass substrate. With the exceptions of sodium tripolyphosphate and sodium oleate, nonionic surfactants were most effective solubilizers and yielded highest soil removal. Effective soil removal and solubilization occurred at concentrations considerably in excess of cmc. (Note results for Decanol-10 EO). The two synthetic anionic surfactants tested were ineffective as solubilizers and as detergents, suggesting that neither had the optimum hydrophobe balance required for this system. Both sodium oleate and sodium tripolyphosphate were effective detergents but displayed essentially no solubilization. This suggests that these agents operate by an entirely different removal mechanism compared to the nonionics tested. Displacement and emulsification are probable routes for the ionic agents. It is also suggested that different mechanisms of soil removal can arise depending on the surfactant type employed.

Conclusions

These data demonstrate for two quite dissimilar systems and evaluation methods, that soil removal for surfactants frequently begins at or near cmc. These findings supplement those of Chandler and Shelberg (2) and the claims of Demchenko (3), and prove that Preston's (10) correlation of cmc and maximum washing power is not the general rule. Maximum soil removal effectiveness occurs when cmc has been exceeded many-fold, the multiple depending upon the surfactant in question.

Because of the correlation between cmc and soil removal, an equation for soil removal could be developed:

$$SR_n = \frac{C_m}{a + b\,C_m},$$

where SR_n = % soil removal minus water blank,

C_m = micellar weight % concentration (total % conc.-cmc), a and b are constants, and 1/b estimates maximum detergency ($SR_{n\,max.}$).

Excellent fit of experimental values to the derived equation for both model systems was found.

Correlation between soil removal and solubilization was shown. Solubilization may be an important mechanism in soil removal for systems comprising fatty and oily soils.

Different types of surfactants vary in their ability to solubilize triolein: Nonionics tested show relatively high capability, anionics and sodium tripolyphosphate solubilize little if any triolein. This shows that since these surfactants are effective soil removers, they operate through different mechanisms: The nonionic by displacement and solubilization, the anionics essentially by displacement: Both can function by emulsifying cohesively bound soil.

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Chromatostrip Analysis of Fatty Acid Derivatives[1]

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Chromatostrips provide a rapid and convenient method of examination of samples by spot tests and by ascending or descending chromatography. Ascending chromatography for the examination of mixtures is carried out on 12 x 140-mm. glass strips coated with 5% starch-bonded silicic acid, while descending chromatography is done on 12 x 200-mm. strips. The 5% starch-bonded silicic acid coatings are resistant to normal handling, may be marked with a soft lead pencil, and may be stored indefinitely for reference. Three detection systems are employed: fluorescent minerals for conjugated unsaturates, fluorescein-bromine for unsaturates, and 2′,7′-dichlorofluorescein for all types of compounds. Positive tests result in characteristic spots when observed under normal or ultraviolet illumination. This permits the classification of components, after separation on the chromatostrips, into the groupings of conjugated unsaturated, unsaturated (or easily brominated), and saturated compounds.

In the past few years the technique of chromatography employing thin coatings of adsorbent bonded to glass surfaces has received considerable attention. A modified technique called "thin layer chromatography" (TLC) was reported by Stahl (1) in 1956. This method was further investigated by this author (2-4), and more recently by Mangold and coworkers (5-7) and others in the field of lipids (8-15), steroids (16, 17), amino acids (18), and other areas (19-24). After noting the success of Morris *et al.* (9) in applying this technique to the analysis of fatty acid derivatives, the conveniently available "chromatostrip" technique of Kirchner, Miller, and Keller (25) was investigated in this same area. The

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chromatostrip technique was first introduced in 1951 (25) and was acknowledged by Stahl (1) as the starting point for his studies (1-4). Its development by Kirchner and Miller (26-28) and others was reviewed by Demole (29) in 1958. In more recent years this technique has been used extensively in our laboratories for the analysis of the constituents of citrus oils (30-37), forage estrogens (38-41), forage saponins and sapogenins (42), and plant polyphenols (43). Fortunately, direct contact with many of these later workers was possible. This provided a considerable background of information and experience, and very little adaptation was necessary for the application of the chromatostrip technique to the analysis of fatty acid derivatives.

Three useful indicator systems were adapted for the classification of fatty derivatives into the groupings of conjugated unsaturated, unsaturated, and saturated compounds. Zinc-cadmium sulfide and zinc silicate phosphors were used to detect conjugated unsaturation, and fluorescein-bromine was used to detect unsaturated or easily-brominated compounds. These systems were described by Kirchner *et al.* (25). All three classes were detected by the elegant dichlorofluorescein method of Mangold and Malins (6). A number of pure compounds of these three classes were examined to demonstrate the utility of this classification system. In addition, the usefulness of the chromatostrip technique for the separation and tentative identification of various fatty acid derivatives was demonstrated by studies with mixtures of selected compounds. The experience and data gained were applied to the analysis of some mixtures obtained in the study of the chemistry of the acids from castor and *Dimorphotheca* oils. These results, the means of obtaining them, and some advantages and limitations of this method will be discussed.

Experimental

Preparation and Storage of Chromatostrips. Chromatostrips are prepared from glass strips ca. 12 mm. wide × 140 or 200 mm. long cut from ordinary (ca. 2 mm. thick) window glass. The strips (clean and dry) are passed through a simple coating device (Fig. 1) that is a modification of the original design of Miller and Kirchner (28). The typical coating is 5% starch-bonded silicic acid. The coating formulation used closely follows that of these authors (28) except Mallinckrodt silicic acid (100 mesh—labeled "suitable for chromatographic analysis by the method of Ramsey and Patterson") is substituted for the Merck reagent grade silicic acid originally specified. The silicic acid (85.5 g.), 4.5 g. of Clinco-15 starch, 1.05 g. of zinc-cadmium sulfide, and 1.05 g. of zinc silicate are well mixed in a dry beaker. Distilled water (172 ml.) is added with stirring to form a smooth slurry. This is slowly heated on top of a steam bath with continuous manual stirring until the temperature reaches 70°C. When this temperature is attained, the thickened paste is cooled at the tap to about 30°C. Judicious additions of water in small portions with stirring yields a thin paste that spreads readily and smoothly. A typical viscosity is ca. 500 centipoise (Brookfield). If the paste is too thin, leakage from the spreader may occur, but the strips usually are satisfactory. On the other hand, too thick a paste formulation lumps and does not spread well. A little experimentation with the paste will reveal a suitable consistency for satisfactory coatings. If the paste thickens during use, it can be thinned at any time by the careful addition of water. If thinned too much, coating of strips should be continued as the paste will drain some water and tend to gel slowly reaching a proper consistency in a few moments.

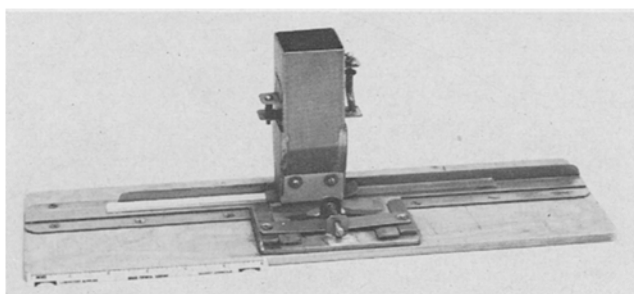

FIG. 1. Modified chromatostrip coater based on original design of Miller and Kirchner (28). (Working drawings are available from the authors.)

The phosphors (zinc-cadmium sulfide and zinc silicate) can be omitted if the use of compounds strongly absorbing ultraviolet radiation in the range 200 to 400 mμ is not anticipated. Such strips can be used with the other two detection reagents; however, if present the phosphors do not materially interfere with these indicators. Strict adherence to the heating procedure and the use of specified types of starch (cf. 28) are essential for satisfactory results.

The coated strips (which should appear smooth and somewhat opalescent when wet) are dried in a forced draft oven for 30 min. at 105°C. Prepared in this fashion the coating layer is less than 0.5 mm. thick. The dried strips are transferred to desiccators and stacked with paperboard separators over NaOH pellets. The strips can be stored in this manner indefinitely and are not damaged by normal handling. They also can be marked with a soft lead pencil. Several hundred strips are prepared at one time by a technician experienced in this technique.

Other coating formulations such as 5 to 30% gypsum-bonded silicic acid (25) or Merck "Silica Gel G"[3] have been briefly examined by spreading thin layers on several glass strips with a notched spatula blade. Recently the apparatus used for coating the glass plates according to Stahl (2) has been successfully used to prepare chromatostrips with "Silica Gel G". This was done by using a glass plate ca. 200 × 700 mm. (2 mm. thick) to which the glass strips (2 mm. thick) were held by the surface tension of a water film. Substituting this for the glass plates (ca. 3.5 mm. thick) usually employed in the Stahl technique (2) permitted the preparation of satisfactory chromatostrips with this instrument.

Compounds. Substances employed as reference standards were obtained from commercial sources (usually as GLC standards) from laboratory stocks, or by standard laboratory preparative methods. All were of adequate purity as judged by the appearance of their chromatograms and by their physical con-

[3] Manufactured by C. Desaga G.m.b.H., Heidelberg, Germany. Obtained from Brinkmann Instruments, Inc.

stants. Reference to the source of mixtures will be indicated as they are discussed.

Development of Chromatostrips. Prior to spotting with the material to be analyzed, a dashed pencil line is drawn across the chromatostrip ca. 1.5 cm. from the bottom. This marks the origin where the solution is applied. Another pencil line (solid) is placed 10.0 cm. away to indicate the completion of development. Other identifying information can then be placed beyond the finish line. The use of exactly 10.0 cm. as a development path permits direct measurement of R_f values and lowers uncertainties due to variation in the extent of development. Samples for analysis are generally made up as 10% solutions in the least polar solvent possible. This reduces spreading of the applied spot. Usual loadings are in the range 100 to 200 μg. applied with an appropriate micropipet. Often however smaller or larger amounts (20 to 1,000 μg.) are used for certain situations, such as precise R_f measurements or preparative scale chromatograms.

For ascending development, the marked and spotted strip is slowly lowered with tweezers into an ordinary 15 × 150-mm. test tube previously charged with 1 ml. of an appropriate developing solvent. The tube is quickly stoppered with a tight-fitting cork, care being exercised to avoid splashing of the solvent onto the chromatostrip. Development begins immediately, and the readily discernible solvent front usually rises by capillary action to the 10 cm. mark in 10 to 15 min. When development is completed the strips are removed and dried in air. Mixtures of polar solvents (such as diethyl ether, ethyl acetate, tetrahydrofuran and methanol) with less polar solvents (such as the petroleum ethers and benzene) have generally proved most valuable for the separation of many fatty acid derivatives. For example, 3:1 or 7:3 Skellysolve F: diethyl ether is the solvent of first choice for the preliminary examination of a mixture of fatty acid esters.

A useful modification of this method is the descending development technique devised by Stanley and Vannier (31). In this modification the solvent, furnished by a filter paper wick dipping into a separate reservoir, flows down a 12 × 200 mm. chromatostrip that is loaded near the top. R_f data can be obtained by operating in a normal fashion with a single solvent system; however, at the expense of R_f data, the strip can be operated as a small, open-faced, chromatographic column. Solvent systems can be changed at will, and fractions can be collected by allowing solvent to drip from the lower end of the strip.

Another modification briefly investigated was the use of the "oversaturation" technique (4, 23). This involved simply inserting a filter paper liner or wick into the solvent supply of the development container. This apparently supplies a higher concentration of solvent vapor around the strip and has been reported (4, 23) to aid reproducibility and speed of development.

Detection of Spots. Following development and air drying, the strips are prepared for spot detection. If fluorescent minerals are used, the strips are first examined under a short wave (254 mμ) ultraviolet lamp.[4] Substances that strongly absorb ultraviolet radiation in this spectral region are detected as dark blue or purple spots due to quenching of the fluorescence of the phosphors (yellow-green at this wavelength). The presence of substances such as conjugated ketodienes or trienes is often suggested by noting a weaker quenching under a long wave (366 mμ) ultraviolet lamp. The ordinary conjugated dienes do not quench the somewhat orange fluorescence in this region, therefore a positive test is indicative of the longer conjugated system. The long wave lamp also seems to give the best results with strips that are sprayed with an 0.2% solution of 2′,7′-dichlorofluorescein in 95% ethanol (6). Almost all classes of compounds show up as yellow fluorescent spots on a somewhat darker background. A few types do not show up or appear as dark brown to purple spots of diminished intensity, but fortunately these are in a minority among the fatty acid derivatives investigated. Nonconjugated olefins and other easily brominated compounds are detected in visible light as white or yellow spots against the pink background of the dye, eosine, formed by spraying the strip with 0.05% aqueous fluorescein and exposing it to bromine vapor (25).

[4] The lamps used are "Mineralight" for 254 millimicrons and "Multi-ray" with "Black Raymaster" tube for 366 millimicrons.

Monitoring of Fractionation Systems. A valuable application of the chromatostrip method is the monitoring of fractionation systems. For example, the effluent from a column chromatographic separation was automatically collected in equal volume fractions. Fixed aliquots (typically 5 microliters) were spotted consecutively on a chromatostrip ca. 5 mm. apart. Without development, the strip was subjected to a suitable detection method. Usually dichlorofluorescein is employed, but the fluorescent minerals are of value with appropriate compounds. The presence of solute fractions is indicated by fluorescent spots, often of increasing and diminishing intensity as a solute peak is traversed.

Results and Discussion

Analysis of Standard Compounds and Mixtures. The results of the ascending chromatostrip analysis of known compounds in some representative solvents using the various indicator systems are presented in Table I. Mixtures of some of these compounds were prepared and separated on chromatostrips demonstrating the utility of the separation and detection methods. These examples are listed in Table II.

The effect of the various solvent systems on R_f values follows the usual pattern expected for adsorption chromatography. The more polar compounds require more polar solvents for movement (elution). The discrepancies in some of the R_f values between Tables I and II require comment. It has been noted (cf. Ref. 7 and others) that increased loadings cause increased R_f values. This was apparently the case in these results. All of the examples in Table I were run at 200 μg. loadings, while those in Table II were run at 200 μg. *total* loading, that is, at ca. 60 to 100 μg. of each component, except for mixture V which contained 200 μg. of each component. This illustrates one of the problems that must be recognized. For careful comparison of R_f values somewhat comparable amounts of given components should be present. This is probably best accomplished by using the minimum amounts compatible with a particular detection method. Reproducibility under similar conditions is

TABLE I

$R_f \times 100$ Values for Some Fatty Acid Derivatives in Various Solvent Systems. Loading 200 γ

Compound	Solvent Mixture					
	A	B	C	D	E	F
Methyl palmitate [a]	62	89				
Methyl stearate [a]	60	89		95	97	81
Methyl oleate [a, c]	56	89				
Methyl linoleate [a,c]	58	88				
Methyl linolenate [a,c]	57	87				
Methyl eleostearate [a, b, c]	49 [d]	82 [d]				
Methyl 9-hydroxystearate [a]		43				
Methyl 12-hydroxystearate [a]		50			87	45
Methyl 9-hydroxy-10, 12-octadecadienoate [a,b,c]		43	67		84	41
Methyl ricinoleate [a,c]		49	72			45
Methyl 12-ketostearate [a]		83			94	55
Methyl 9-keto-10, 12-octadecadienoate [a, b, c]		61				
Monopalmitin [a]					45 [d]	35 [d]
Monostearin [a]				20	35	31
Distearin [a]				79	93	58
Monoricinolein [a]		0		8	21	26
9-Hydroxystearic acid [a]		14	55			27
12-Hydroxystearic acid [a]		16	62			27
9-Hydroxy-10,12-octadecadienoic acid [a,b]		16	46			27
Ricinoleic acid [a]		22	58			27
Ricinelaidic acid [a]		22	64			28

Solvent Mixtures:

A. 90% Skellysolve F—10% diethyl ether (v/v).
B. 70% Skellysolve F—30% diethyl ether (v/v).
C. 50% Skellysolve F—50% diethyl ether (v/v).
D. 75% Benzene —25% diethyl ether (v/v).
E. 50% Benzene —50% diethyl ether (v/v).
F. 85% Benzene —15% methanol (v/v).

[a] 2',7'-Dichlorofluorescein indicator.
[b] Quenching of fluorescent minerals.
[c] Fluorescein-bromine indicator.
[d] Major spot, minor impurity noted.

TABLE II

$R_f \times 100$ Values for the Components of Some Mixtures of Fatty Acid Derivatives in 7:3 Skellysolve F: Diethyl Ether.
Loading: I-IV, 200 γ; V, 600 γ

Mixture	Compounds	Indicator		
		Ultraviolet Quenching	Dichlorofluorescein	Fluorescein-Bromine
I:	Methyl oleate	....	91	89
	Methyl 12-ketostearate	....	75	....
	Methyl 9-hydroxy-10,12-octadecadienoate	36	39	36
II:	Methyl stearate	....	91	....
	Methyl 9-keto-10,12-octadecadienoate	59	61	62
	Methyl 9-hydroxy-10,12-octadecadienoate	38	40	38
III:	Methyl stearate	....	92	....
	Methyl 12-ketostearate	....	77	....
	Methyl 9-hydroxystearate	....	44	....
IV:	Methyl palmitate	....	93	....
	Methyl ricinoleate	....	45	46
V:	Methyl stearate	....	91	....
	Methyl 9-keto-10,12-octadecadienoate	62	59	63
	Methyl ricinoleate	....	47	50

adequate (cf. Fig. 2), but there is no substitute for checks of R_f values by running suitable controls. This has been adopted as standard practice and tends to remove uncertainties due to experimental variations.

It must be stressed that the results presented here are relative "R_f values" applicable only to a certain system manipulated in a particular way. Limited tests yielding varying results with several modifications including the "oversaturation" method (4, 23) offer convincing evidence that R_f values must be compared only within a given set of experimental conditions. For instance, the "oversaturation" technique offers an advantage of faster development in shorter times (ca. 30%), but somewhat lower R_f values are obtained than when operating without the filter paper liner. If any given condition is maintained, however, reproducibility is adequate. For cases where utmost confidence is required, the technique of mixed chromatograms (27) may be of value when comparing chromatographic behavior of knowns and unknowns.

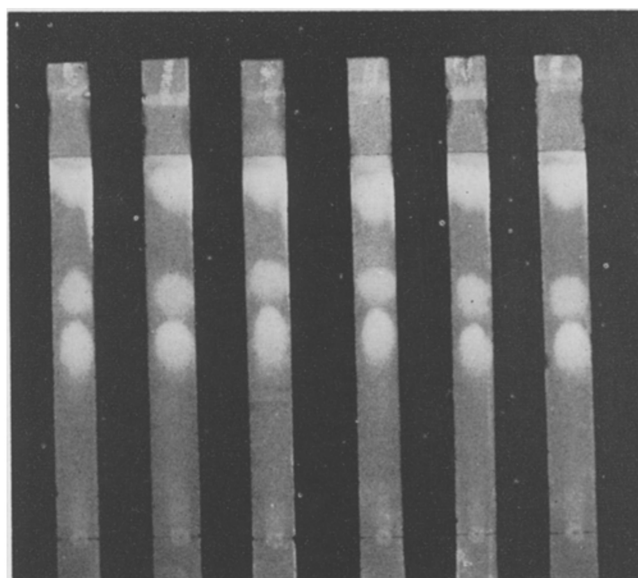

FIG. 2. *Reproducibility Test.* Strips were developed consecutively with 7:3 Skellysolve F: diethyl ether after loading with 200 γ each of methyl stearate (upper spot), methyl 9-keto-10,12-octadecadienoate (middle spot), and methyl ricinoleate (bottom spot). Indicator: 2',7'-dichlorofluorescein. Illumination: 366 millimicron light.

The suitability of the detection methods is outlined in Table II. In every case the conjugated dienes are detected directly by fluorescence-quenching, and as olefins with the fluorescein-bromine test. The other easily-brominated components are all revealed by this latter test, while all types are detected with the dichlorofluorescein indicator.

In tests of the limits of sensitivity of these detection methods it has been possible to detect as little as 5 to 10 μg. of various materials, after development to R_f values of about 0.5, both with dichlorofluorescein and fluorescence-quenching when applicable. One μg. can be detected easily when put on in a concentrated spot (ca. 1 mm. diameter) as in fractionation monitoring. No such limit was established for the fluorescein-bromine test, but experience suggests that this test method is somewhat less sensitive than the other two.

Fractionation Monitoring. Typical results obtained when monitoring a column chromatographic separation are shown in Fig. 3. Fractions of a separation of a mixture from the partial alcoholysis of a castor glyceride were spotted on a plain strip and sprayed with dichlorofluorescein. The limits of the eluted fractions are quite clearly indicated. This permits a rapid detection of the solute peaks which can then

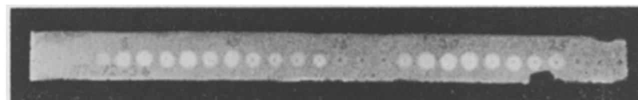

FIG. 3. *Fractionation Monitoring.* Five microliter aliquots applied without development. Indicator: 2',7'-dichlorofluorescein. Illumination: 366 millimicron light.

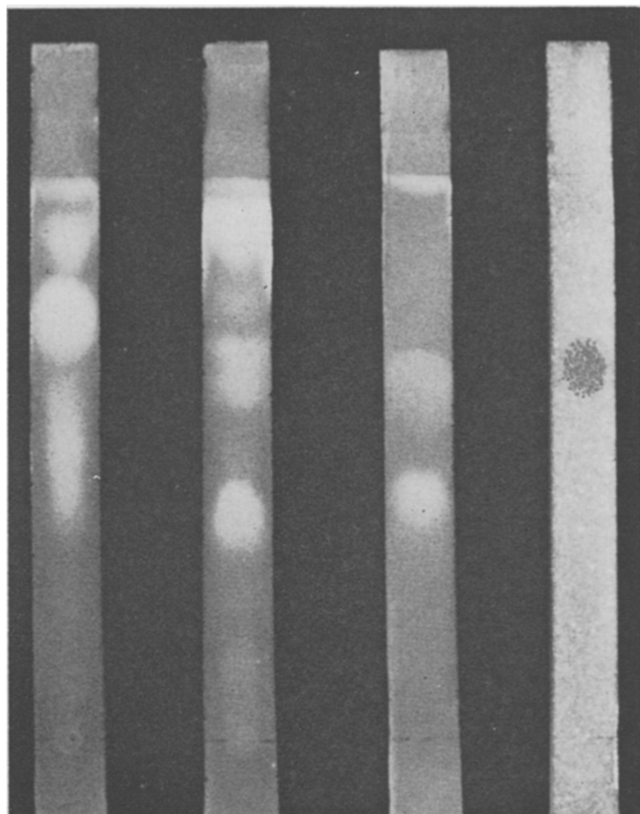

FIG. 4. *Reaction Product Examination.* Strips from left to right loaded with 500 γ of: 1. Crude, isomerized methyl ricinoleate. 2. Crude, hydrogenated methyl dimorphecolate. 3, 4. Product from reaction of α-naphthyl isocyanate with methyl 12-hydroxystearate. Indicators: 1–3, 2′,7′-dichlorofluorescein. 4, fluorescent minerals. Illumination: 1–3, 366 millimicron light. 4, 254 millimicron light.

be consolidated and isolated. The noted sensitivity of approximately 1 μg. means that in a 10 ml. fraction as little as 2 mg. will be detected, if a 5 μl. aliquot is used. This method offers the added advantage of rapid examination of eluates for extent of fractionation. The other indicator systems or combinations thereof may be used in applicable instances.

Analysis of Unknown Mixtures. The ascending chromatostrip method is routinely applied to the analysis of mixtures of unknown composition. A few representative examples will serve to demonstrate the use of the method. Fig. 4 is a photograph of the chromatostrips used, and these will be discussed sequentially from left to right.

The crude product obtained in high yield from the Raney-nickel catalyzed isomerization of methyl ricinoleate to methyl 12-ketostearate according to Hanford *et al.* (44) or Colonge and Guyot (45) was analyzed in the solvent system 7 : 3 Skellysolve F : diethyl ether. The chromatostrip showed spots with R_f values of 0.89, 0.77, and 0.55 overlapping 0.45 when viewed at 366 mμ after spraying with ethanolic dichlorofluorescein. Tests for unsaturation were negative or uncertain. Speculation regarding the likely origin of the spots representing the various compounds is possible by a consideration of likely reactions and comparison to the values in Table I. Considering the possibility of direct hydrogenolysis, or dehydration and reduction, or of isomerization alone, it is not unrealistic to assume the presence of methyl stearate ($R_f = 0.89$), methyl 12-ketostearate ($R_f = 0.77$), and possibly methyl 12-hydroxystearate ($R_f = 0.55$). The overlapping spot at $R_f = 0.45$ may arise from unreacted methyl ricinoleate (unlikely, since no unsaturation was noted) or from interesterified products likely to occur in the presence of the basic Raney-nickel at this temperature (220°C.).

The results from an examination of the residue from the mother liquors of the crystallization of methyl 9-hydroxystearate after the reduction of methyl dimorphecolate with hydrogen and platinum oxide in methanol according to Smith *et al.* (46) seemed to fit a similar pattern. Again no unsaturation was evident, and chromatostrip analysis with dichlorofluorescein indicator revealed several spots of R_f 0.92, 0.77, 0.67, and 0.42. Partition by GLC analysis on a silicone rubber stationary phase also showed several overlapping peaks. Both the R_f values and GLC retention times could be correlated with the methyl esters, i.e., stearate, ketostearate, and hydroxystearate, expected from hydrogenolysis, isomerization, and reduction reactions. A number of side reactions apparently occur in this reduction system, and these results agree with the authors' report (46) that this was a somewhat unsatisfactory method.

A final example involves a purity check. A strip was loaded with recrystallized material from the reaction of α-naphthyl isocyanate and methyl 12-hydroxystearate and developed as usual in 7 : 3 Skellysolve F : diethyl ether. Examination of the strip under 254 mμ light showed one spot ($R_f = 0.68$) due to absorbance (and fluorescence-quenching) by the aromatic moiety of the α-naphthylurethane. Even more enlightening, however, was the overspray with dichlorofluorescein which showed up both the main product ($R_f = 0.68$) and the starting methyl 12-hydroxystearate ($R_f = 0.44$). This showed that crystallization was not a satisfactory means of purification, at least with the solvents used, and the information gained was employed in carrying out a column chromatographic purification of the material. The latter separation was monitored by the technique mentioned earlier.

Summary

The chromatostrip technique provides a simple, inexpensive, relatively reproducible and rapid system of microanalysis of mixtures. Further, the strips and indicators are useful in monitoring fractionation systems. The coatings are rugged enough to be handled and stored, and hard enough to be marked with a soft lead pencil. This is a very worthwhile feature of the system. Labeling of the strips is simplified and the chromatograms can be retained indefinitely. As scores or even hundreds of strips can be kept on hand, the method is always readily available for immediate use. A single strip analysis can be carried out in as little as 10 to 15 min., and often 6 to 10 analyses are run in an overlapping sequence. In addition, some clue as to the character of the components is obtained by their R_f behavior in various solvents and their response to different indicator systems. The older criticisms (cf. 1) of the chromatostrip technique, such as difficulty of preparation and lack of reproducibility of the coatings, have not been apparent in these studies. It was found recently that the Merck "Silica

Gel G" used in the Stahl modification (2) gives substantially better resolution and spot tightness with some lipid mixtures than does the Mallinckrodt silicic acid. However, the gypsum-bonded layers are extremely fragile on the glass strips employed. Thus its use in this form has been given only a cursory examination. It is felt that the hardness of the starch-bonded materials offers a substantial advantage, and work with similar silica gels with starch binder is in progress.

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• *Letter to the Editor*

Mass Spectrometry and Lipid Research

TWO DEVELOPMENTS in commercial equipment for mass spectrometry are of significance to lipid research: a) heated inlet and ion source systems for use with "high boilers" and b) rapid photographic registration of mass spectra. Preliminary results from our laboratory demonstrate the applicability of these advances to problems of determining the position of double bonds in monoenes and of identifying volatiles from reverted soybean oil.

Dinh-Nguyen, Ryhage, and Stenhagen have shown that the position of the double bond in methyl petroselinate and oleate may be located by first deuterating the double bond (1). This deuteration step is necessary since the initial position of the double bond (except for the α, β locations) is not reflected in the mass spectra of the nonoene isomers (2).

With the use of a heated (200°C.) sample inlet, ion source, and flight tube, the mass spectrum of methyl 6,7-dideutero stearate was also recorded in our laboratory but with a "Time-of-Flight"-type spectrometer. Shown in Fig. 6 of a paper appearing in this issue (3) are strip chart recordings of the spectra for methyl 6,7-dideutero stearate and for a mixture of stearate and dideutero stearate. Mass peaks at 298 and 300 (parent peaks) correspond to normal methyl stearate and methyl dideutero stearate ions, respectively, as well as do the fragment ions at 129 $[-(CH_2)_5COOCH_3^+]$ and 130 $[-CHD-(CH_2)_4COOCH_3^+]$ and at 143 $[-(CH_2)_6COOCH_3^+]$ and 145 $[-CHD-CHD-(CH_2)_4COOCH_3^+]$. The fragments differing from each other by one and two mass units are critical in determining the position of the deuterium and, hence, in ascertaining the original position of the double bond. These data confirm the prior observation of workers in Sweden, who used a magnetic scanning-type spectrometer (1).

In the course of identifying hydrocarbons in volatiles of reverted soybean oils (4), a preliminary analysis was made on Phillips hydrocarbon mixture No. 37. This analysis was achieved by gas chromatography on a polypropylene glycol column at −20°C. (3), Fig. 11. Effluents were monitored by the mass spectrometer in a tandem arrangement described by Gohlke (5) in order to identify the components as they emerged. In the chromatogram of the Phillips mixture particular interest resides in the last peak eluted which appears to be a single component. Actually, it is composed of two components, *cis*-butene-2