during transport the plasticity may disappear completely. Certain fat compositions are more sensitive to this than others. By means of worksoftening determinations the plasticity can be followed.

Shortenings. Especially when these fats must be worked intensively, not only a correct hardness before kneading, but also after kneading is important.

Spreadability of Butter and Margarine. In an earlier publication (5), the correlation between the yield value and the spreadability on bread was mentioned. This correlation is only valid if the products have all more or less the same worksoftening (70–75%). If this is not the case, the situation is quite different, which becomes especially manifest if the spreadability of butter and margarine is compared. Butter mostly has a lower worksoftening (50–55%) as compared with margarine (70–75%). If the hardness before kneading of the two products is the same, test panels will mostly assess margarine as being more easily spreadable than butter, since the hardness of margarine decreases to much lower values during spreading than butter. In such cases in which the spreadability of samples with different worksoftening values must be compared, C_u and C_w as such are less useful.

Excellent results are obtained if a Spreadability Index (S.I.) is calculated from C_u and C_w.

$$S.I. = C_u - 0.75\ (C_u - C_w)$$

The factor 0.75 was derived by comparing the results obtained with the apparatus with those of panel tests. In this way a high correlation coefficient (>0.95) was found between S.I. and the assessment by panels of housewives.

REFERENCES

1. Haighton, A. J., Fette Seifen Anstrichmittel *65*, 479–482 (1963).
2. Temple, M., van den, J. Colloid Sci. *16*, 284–296 (1961).
3. Lucassen-Reynders, E. H., "Stabilization of Water-in-Oil Emulsions by Solid Particles," Thesis, Utrecht 1962, Chapter IV.
4. American Society for Testing Materials, 5-25 and D 217-52 T, Philadelphia, Pa.
5. Haighton, A. J., JAOCS *36*, 345–348 (1959).

[Received May 11, 1964—Accepted August 10, 1964]

A Light Test to Measure Stability of Edible Oils

HELEN A. MOSER, C. D. EVANS, J. C. COWAN and W. F. KWOLEK,[2]
Northern Regional Research Laboratory,[3] Peoria, Illinois

Abstract

The effect of light on the flavor of edible oils and of various fat-containing foods is reviewed to show its importance in food studies and the need for a method of evaluation. Such a test, in which fluorescent light is used in an easily assembled unit, has been developed, and the parameters for its use have been determined. Identical samples of soybean oil exposed on 10 different days and organoleptically evaluated show the method to be reproducible with a standard deviation of 0.79 with a scoring system of 1–10. This method was then applied to soybean, cottonseed, safflower and hydrogenated-winterized soybean oils, and a light-exposure value was determined for each oil based on a comparison with accelerated storage procedures ordinarily used. Advantages of this light test over current procedures are the short time required for completion, the reduction of variation by a controlled light source, reproducibility of results and its adaptability to related food products.

Introduction

THE DELETERIOUS EFFECT of light on the flavor stability of edible oils and various fat-containing foods is well known (5–9, 12–14, 16,17). However much of the work was done between 1930 and 1947; since then, few studies have been reported (21,22). The increased consumption of soybean oil, as well as the recent emphasis on the use of liquid oils in the diet, has renewed interest in the subject. In the fall of 1963 a survey of five local supermarkets showed that for the 15 brands of salad oil represented (5 cottonseed, 4 corn oil, 4 safflower, 1 soybean, 1 peanut) all were bottled in clear glass. Some of these oils previously had been marketed in brown glass bottles or in cans only.

In the past, grocery stores were small and products were received in corrugated cartons; a few items were removed at a time and placed on the shelves only as needed. This resulted in a relatively quick turnover (3). The small store of the past was not as intensely lighted as the modern supermarkets and the problem of oils and other food products developing off flavors while on the shelf because of exposure to bright fluorescent lighting arises under today's methods of merchandising.

Coe and LeClerc (4,9) studied the effect of light on the peroxide development of oils and showed that oils protected by black or green wrappings developed no rancidity even though the peroxide value was high. Golumbic et al. (11,12) investigated light reversion in fats exposed to IR and to UV radiation. IR exposure produced rapid reversion that resulted in well-defined flavors, such as grassy or haylike, easily recognized and described by all; the UV exposure produced samples difficult to describe and characterized by a drying sensation in the roof or back of the mouth. McConnell and Esselen (16,17) conducted light tests and found amber glass, which excluded most of the incident light below 500 mμ, effective in retarding off-flavor development in edible oils exposed to diffused light. However, amber glass afforded less protection when samples were exposed to direct sunlight. Gudheim (15) also found amber glass more effective than green, blue or opal glass in retarding off-flavor development in shortenings and liquid oils, and he reported that flavor and odor changes were not characteristic of rancid fat. Romani and Valentinis (21) showed that both the Kreis and peroxide values of fresh and aged edible oils increased on exposure to light. Higher viscosities and increased acetyl numbers resulted when Ryspaev (22) exposed fats to UV radiation.

Research on light effects has not been limited to

[1] AOCS Bond Award Honorable Mention, Fall 1963.
[2] Biometrician, ARS Biometrical Service, stationed at Northern Laboratory.
[3] A laboratory of the No. Utiliz. Res. & Dev. Div., ARS, USDA.

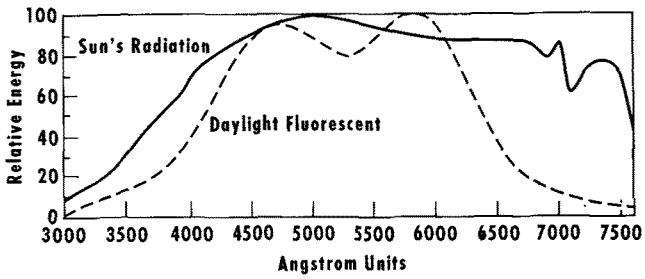

FIG. 1. Spectral energy distribution of natural and artificial light.

edible oils. Many other food products either high in oil content or prepared with oil are subject to light deterioration and are being studied. Musco and Cruess (20) studied the shelf life of walnut meats and walnut oil and found that both were susceptible to light deterioration and that the oil deteriorated much more rapidly than the meats. Manufacturers of potato chips have been concerned with flavor for a long time and recognize light as a contributing factor to off-flavor development. Today, as a result of many laboratory investigations, potato chips are universally packaged in cardboard cartons or in yellow cellophane, opaque and foil bags (25). Work by Anderson (2) indicates that exclusion of light below approx 490 mμ is necessary to protect milk from light-induced flavor. Samuelsson and Thorne (23) believed that the "sunlight" flavor of milk may be caused by the formation of certain aldehydes, and Wildbrett (28) reports that sunlight flavor is reduced by the use of brown bottles or paper cartons. However, the increased use of wax- or plastic-coated cartons has not solved the problem since a 1962 publication of Dunkley et al. (10) states that milk in cartons exposed to fluorescent light developed off flavors in four hr. Light flavor could be detected in milk stored in a clear glass bottle after 20 min exposure; in an amber glass bottle after five hr, and in various fiberboard cartons after 1–14 hr.

Scott (24) reported that the stability of medicinal white oil was affected by light and that when treated with oxidation inhibitors, it was not stable to light even though stable to heat. He also pointed out the need for an accelerated laboratory procedure which "acceptably simulates the action of sunlight and assesses deterioration in a well-defined manner." Our paper reports the development of a light-stability test for edible oils using fluorescent light.

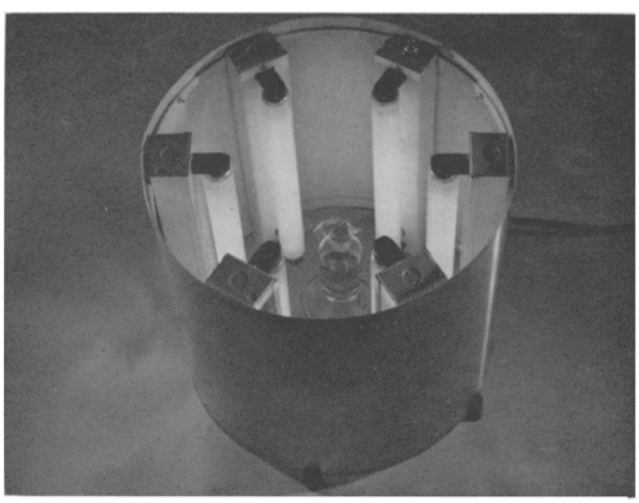

FIG. 2. Light exposure apparatus used in measuring the stability of edible oils.

TABLE I

Effect of Daylight and Fluorescent Light on the Flavor Stability of Soybean Oil

Light source	Exposure (hr)	Flavor score	Peroxide value
North window	6	5.9	1.2
Fluorescent, 6 tubes	1	5.7	1.5
Control	0	7.7	0.4

Experimental

Because the term "exposure to light" can mean many things, i.e. exposure to north or south window light, to direct sunlight, diffused light or light at a lab bench, fluorescent light was investigated. By using fluorescent light, the light source can be standardized and the variation within a single day and between days can be eliminated. The initial work was done with two desk lamps, each equipped with two 18-in., 15-w daylight fluorescent tubes. These were adjusted so that the bottle of oil placed between them received approx the same amount of light as from a north window on a bright day. Daylight fluorescent bulbs have a color temp of 6500K, which is similar to the combination of sunlight and skylight of a north window. The spectral energy distribution over the range of 300–750 mμ for daylight and for daylight fluorescent lamps is also similar and is shown in Figure 1 (26).

A comparison of samples exposed for six hr in both areas was made, and while the results of this test were comparable, the time element was a drawback. It was decided to increase the illumination and thus shorten the time of exposure. The light apparatus used in the subsequent tests is almost identical with one designed and used by Gudheim (15) in shortening tests and with one used by Mounts and Dutton (19) in plant-growth studies. Figure 2 shows the apparatus constructed with six 15-in., 14-w daylight tubes mounted inside of a 17.5-in. diam stainless-steel drum, 17.5 in. high. The interior of the drum was painted white to reflect light from the backside of the fluoresecent tubes. The drum was mounted on 1.25 in. feet, and because the top is open, air circulates freely. The exposure apparatus was operated in a taste panel room where the temp is always constant at 78F. Although temp of the sample increased slightly during exposure, flavor differences due to this factor were not observed. This apparatus shortened the time needed for testing from 6 hr to 1 hr. A comparison of flavor scores and peroxide values is given in Table I.

The oils tested for flavor stability to light included cottonseed, soybean, safflower and hydrogenated-winterized soybean (HWSB). Both commercially and laboratory-deodorized samples were tested. While it is assumed that most commercially prepared samples contain stabilizers, the quantities added are not

TABLE II

Effect of Light on Flavor Scores and Peroxide Values of Commercially Deodorized Samples

Exposure (hr)	Soybean	Cottonseed	Safflower	HWSB "A"[a]	HWSB "B"
0	8.0 (0.5)[b]	6.7 (0.5)	6.7 (2.4)	7.7 (0.4)	6.9 (0.4)
0.5	6.0 (1.9)	6.0 (1.0)		6.7 (0.8)	5.6 (0.7)
1	5.8 (2.5)	5.5 (1.1)		5.0 (1.7)	5.1 (1.3)
2	4.6 (2.8)	5.2 (1.6)	6.2 (3.5)	4.5 (2.5)	4.9 (1.2)
3		5.1 (2.1)		4.2 (2.9)	
4	4.4 (2.5)	4.5 (2.0)	5.8 (5.2)	4.0 (2.9)	4.7 (2.2)
6	4.2 (2.9)		4.9 (5.1)	4.0 (3.7)	4.6 (2.2)
8			4.8 (6.0)		4.3 (2.3)
40			3.8 (9.8)		
AOM[c]	11.0	27.7	22.7	2.5	12.4

[a] HWSB: Hydrogenated-winterized soybean oil.
[b] Figures in parentheses are peroxide values determined at time of evaluation.
[c] Peroxide values after 8 hr under AOM conditions.

TABLE III

Effect of Light on Flavor Scores and Peroxide Values of Laboratory-Deodorized Samples

Exposure (hr)	Soybean	Cottonseed	Safflower	HWSB[a]
0	7.9 (0.4)[b]	7.9 (0.4)	8.2 (1.2)	6.3 (1.0)
0.5	6.8 (0.9)	7.4 (0.8)		5.8 (1.6)
1	5.7 (1.5)	7.0 (1.1)	7.4 (2.3)	5.6 (1.4)
2	5.6 (1.7)	5.6 (1.5)		5.3 (2.0)
3	5.3 (1.8)	5.7 (2.0)		
4			7.1 (3.9)	
6			7.0 (3.5)	
8		5.6 (5.1)	6.8 (4.0)	
16			6.6 (5.7)	
24			5.9 (6.3)	
40			5.9 (7.8)	
AOM[c]	26.7	29.4	54.5	36.1

[a] HWSB: Hydrogenated-winterized soybean oil.
[b] Figures in parentheses are peroxide values determined at time of evaluation.
[c] Peroxide values after 8 hr under AOM conditions.

known. The laboratory-deodorized samples did not contain antioxidants or stabilizers. The samples were bottled the same as for the usual accelerated storage tests: 150 ml of oil in an 8-oz clear glass bottle closed with a cellophane-covered cork stopper. The exposure time varied from 0.5–40 hr. The oils were also stored at 60C for four days, and results after storage were compared with those from the light-exposure test.

The oils were organoleptically evaluated following the procedure established at this laboratory (18). Light-exposed samples were paired with control samples (not exposed) and evaluated by a panel of 18 trained judges. The oils were warmed to 55C, and when color differences were apparent, red lights were used in the taste panel room to minimize color. A 10-point scoring system was the basis for judging quality and intensity of flavor. Analysis of variance and the F test were used to test the sample means for differences.

Immediately after exposure peroxide values were determined by the Wheeler method (27). Stability was also measured by determination of the peroxide value after holding the oils for eight hr under Active Oxygen Method (AOM) conditions.

Results and Discussion

Fluorescent light produced definite flavor changes in edible oils, and these changes were readily recognized by the taste panel. A flavor described as grassy or green was pronounced and in later evaluations the term "light-struck" was used to describe samples exposed to light. This light-struck flavor seems to be combined with a mouth sensation described by many as astringent. Because of this added sensation, the term light-struck distinguishes these oils from oils having a grassy flavor caused by something other than light exposure.

Exposures of 0.5–1 hr produced significant changes both in flavors and peroxide values of the oils. Judging from the data in Tables II and III, the flavor of almost all oils is affected to the greatest degree in the first 30–60 min exposure and from then on the change is gradual. The same effect appears to be true for the development of peroxide values. There is little difference in the stability of the different oils. Even though one would expect HWSB oils to be more stable, the samples used behaved much like salad oils. There was a noticeable difference in the light stability of the laboratory-deodorized cottonseed and safflower oils compared to the commercially deodorized samples. Commercial samples differ from lot to lot in their stability to light.

TABLE IV

Comparison of Flavor Scores from Oven-Storage and Light-Exposure Tests

Oil sample	Storage 60C, 4 days	Light exposed	LEV,[a] hr
Commercially deodorized:			
Soybean	5.7 (2.5)[b]	5.8 (2.6)	1
Cottonseed	4.7 (9.9)	4.5 (2.0)	4
Safflower	5.6 (9.8)	5.8 (5.2)	4
HWSB "A"	6.1 (1.3)	6.5 (2.3)	0.75
HWSB "B"	5.4 (1.6)	5.1 (1.3)	0.75
Laboratory deodorized:			
Soybean	5.8 (2.9)	5.7 (1.5)	1
Cottonseed	5.1 (11.6)	5.6 (5.1)	8
Safflower	5.0 (13.4)	5.9 (6.3)	24

[a] Light equivalent values.
[b] Figures in parentheses are peroxide values determined at time of evaluation.

TABLE V

Comparison of Stability Tests for Commercially Prepared Soybean Oil

Test	N	Mean	S.D.	C,[a] %
Flavor score:				
4 Days, 60C	79	5.9	1.39	23.6
2-hr light exposure	100	5.5	0.79	14.3
Peroxide value:				
4 Days, 60C	8	2.46	0.22	8.9
2-hr light exposure	10	2.78	0.20	7.2
AOM, 8 hr	14	13.52	1.50	11.1

[a] Coefficient of variation (S.D./Mean).

AOM peroxide values are often used to predict oil stability. An oil with a low AOM value, such as 5.0 (after 8-hr AOM conditions), should be stable and perform well in its various uses. However, sometimes no relationship to flavor stability can be shown. In Table III the AOM value of the laboratory-prepared sample of soybean oil is twice that of the commercial sample, which gives reason to predict that the commercial sample is more stable. However, actual results show that these two samples give similar results on both oven storage (60C) and light exposure. The commercial safflower sample should be twice as stable as the laboratory sample as far as AOM values are concerned. While there was little difference in oven storage (60C) results, the light-exposure tests proved the laboratory sample to be six times more stable.

Work at this laboratory showed that the flavor of soybean oil dropped significantly after four days' storage at 60C; therefore, this test could be used to measure its stability. The light test can be used in the same way. Light equivalent value (LEV) is the number of hr necessary to give a flavor score equivalent to that obtained after four days' storage at 60C, and this value can be used as a test to predict stability. The LEV for various oils is shown in Table IV. Because exposure to light is much more destructive than oven storage, quality testing can be performed more simply and more quickly. For example, the stability of a soybean oil sample can be judged in one hr instead of either eight hr by the AOM method or four days by the oven-storage method. Development of peroxides in light-struck oils did not always correspond to those developed in oven storage. Peroxide values for soybean oil exposed to light were comparable to those developed in 60C storage, but the values for safflower and cottonseed oils showed poor agreement.

The precision of this method was determined by exposing samples from the same lot of commercially processed soybean oil in the light-exposure apparatus on 10 different days and presenting these exposed samples, with a control, to the taste panel for evaluation.

TABLE VI

Effect of Container on Light-Exposed Soybean Oil

Container	Exposure hr	Flavor score	Peroxide value
Brown glass	2	7.9	0.85
Clear glass	2	5.5	2.24
Translucent plastic	2	5.1	2.46
Control	0	8.1	0.49

The sample means ranged from 5.1–5.8, but day-to-day variation was not significant. Sandard deviations were homogeneous and of ca. the same order as in the past. The pooled standard deviation for the 10 days was 0.79.

This light test may be compared with other stability tests by examining the relative standard deviations or coefficients of variation (Table V). Both organoleptic evaluation and peroxide values vary less than methods now used. When compared with AOM values, the variation of the organoleptic test is only slightly more than the AOM method for testing stability. This degree of precision, plus the shorter time required for the test, appears favorable to most testing programs.

In France, peanut oil is being marketed in disposable, translucent plastic bottles (1). The light test was used to compare the effect of container on the stability of soybean oil. The 1-liter translucent plastic bottle from France, a 1-qt clear glass bottle and a 1-qt brown glass bottle were filled with soybean oil and exposed to light for 2 hr. The results are shown in Table VI. The oil stored in the brown glass bottle was scored only slightly lower than the control sample, which came from a 5-gal tin, whereas the oils stored in both the clear and the plastic bottles were scored significantly lower than either the control or the oil stored in brown glass.

ACKNOWLEDGMENTS

Preparation of the samples by Particia Cooney and David McConnell; information concerning fluorescent light systems from William Tometich, Central Illinois Light Co.

REFERENCES

1. Anon. Extrait de la Revue, "Emballages" No. 201, January-February 1962.
2. Anderson, K. P., Intern. Dairy Congr., 15th (London) *3*, 1746–1753 (1959); C.A. 4946 (1960).
3. Carpenter, D. C., Ind. Eng. Chem. *25*, 932–934 (1933).
4. Coe, Mayne R., U.S. 1,880,813 (1932).
5. Coe, Mayne R., Oil & Soap *15*, 230–236 (1938).
6. Coe, Mayne R., *Ibid. 16*, 146–147 (1939).
7. Coe, Mayne R., *Ibid. 18*, 227–231 (1941).
8. Coe, Mayne R., *Ibid.* 241–247 (1941).
9. Coe, Mayne R., and J. A. LeClerc, Ind. Eng. Chem. *26*, 245–248 (1934).
10. Dunkley, W. L., J. D. Franklin and R. M. Panghorn, Food Technol. *16*, 112–118 (1962).
11. Golumbic, C., C. J. Martin and B. F. Daubert, Oil & Soap *23*, 187–191 (1946).
12. Golumbic, C., C. J. Martin and B. F. Daubert, *Ibid. 23*, 360–361 (1946).
13. Greenbank, G. R., and G. E. Holm, Ind. Eng. Chem. *25*, 167–168 (1933).
14. Greenbank, G. R., and G. E. Holm, *Ibid. 33*, 1058–1060 (1941).
15. Gudheim, A. R., Oil & Soap *20*, 197–201 (1943).
16. McConnell, John E. W., and W. B. Esselen, Jr., *Ibid. 23*, 369–374 (1946).
17. McConnell, John E. W., and W. B. Esselen, Jr., JAOCS *24*, 6–13 (1947).
18. Moser, Helen, A., H. J. Dutton, C. D. Evans and J. C. Cowan, Food Technol. *4*, 105–109 (1950).
19. Mounts, T. L., and H. J. Dutton, Efficient Production of Biosynthetically Labeled Fatty Acids, JAOCS, *41*, 537–539 (1964).
20. Musco, D. D., and W. V. Cruess, J. Agr. Food Chem. *2*, 520–523 (1954).
21. Romani, Bruno, and Gastone Valentinis, Boll. Lab. Chim. Provinciali (Bologna) *10*, 358–363 (1959); CA 2948 (1961).
22. Ryspaev, S. R., Tr. Kirgizsk. Gos. Med. Inst. No. 8, 260–264 (1956); CA 5124 (1960).
23. Samuelsson, E. G., and K. E. Thorne, Intern. Dairy Congr., 15th (London) *1*, 488–496 (1959); CA 4947 (1960).
24. Scott, C. G., in "Gas Chromatography," ed. R. P. W. Scott, Butterworths, Washington, 1960, chap. 26.
25. U.S. Dept. Agr., "Manufacture of Potato Chips," Bur. Agr. Ind. Chem., Washington, D.C., 1945. 2 pp. Processed.
26. Weitz, C. E., "General Electric Lamps," General Electric, Cleveland, 1950, pp. 38–39, 57–60.
27. Wheeler, D. H., Oil & Soap *9*, 89–97 (1932).
28. Wildbrett, Gerhard, Molkerei-Käeserei-Ztg. *15*, 1783 (1959); CA 7917 (1960).

[Received March 16, 1964—Accepted August 6, 1964]

Mustard Seed Processing: Improved Methods for Isolating the Pungent Factor and Controlling Protein Quality[1]

G. C. MUSTAKAS, L. D. KIRK, V. E. SOHNS and E. L. GRIFFIN, JR.,
Northern Regional Research Laboratory,[2] Peoria, Illinois

Abstract

A modified cooking and extraction process for mustard seed is reported in which the pungent factor, allyl isothiocyanate, is separated from the seed to yield triglyceride oil and protein meal. Although removal of the pungent factor from the oil and meal products was previously reported, investigations were continued to develop critical improvements in the process. A reduction in conversion time, combined with steam stripping and shorter heating preiods, resulted in quantitative recovery of the essential oil and in improved protein quality, as measured by the basic amino acids. Biological testing with rats showed the processed meals to be free of toxic and goitrogenic factors and to be well utilized nutritionally. Preliminary estimates indicate that process costs are nearly the same as for a comparable soybean plant.

Introduction

MODIFIED COMMERCIAL OILSEED techniques, such as those used in processing soybeans and cottonseed, have been applied successfully to mustard seed at this Laboratory. In previous studies (7,8) the basic method was developed. The integrated enzymatic and lipid extraction process leads to three products—triglyceride oil, a palatable protein meal and the pungent factor, allyl isothiocyanate. This paper presents new studies which obtained significantly improved separation of the pungent factor to give near theoretical recovery, along with process modifications which improved oil meal quality.

[1] Presented at AOCS Meeting in Toronto, 1962.
[2] No. Utiliz. Res. & Dev. Div., ARS, USDA.

Materials, Methods, and Equipment

In these studies, oriental mustard seed, *Brassica juncea,* was obtained from two lots of seed grown in Montana and received during 1960 and 1961, respectively. The seed lots averaged 7% moisture, 38.3% oil, 22.5% protein and approx 10% hull content. Glucoside content, expressed as converted allyl isothiocyanate, averaged 0.7% moisture-free basis. Commercial grade *n*-hexane was used as solvent in the filtration-extraction.

Allyl isothiocyanate was determined by Wetter's procedure (10). Purity of the essential oil was determined by a modification of procedure in which an aliquot of the oil in ethanol was added directly to the ammoniacal silver nitrate solution. Purity of the allyl isothiocyanate was also analyzed by GLC on a Beckman GC-2A (6,10) packed with Apiezon-L on Celite (40–60 mesh) with a nitrogen flow of 60 ml/min at a temp of 115C. Crude fat was determined by extracting with pentane-hexane in a Bütt extractor for six hr and drying overnight in a vacuum oven at 80C. Amino acid analyses were obtained by hydrolyzing