

the units of the main chain are split once, and that the other half of the units of the main chain, or those at the points of branching, are not split at all.

Experimental

Periodate Oxidation.—Following the method of Hirst, a 400-mg. sample of guaran was dissolved in 100 ml. of potassium chloride solution (5 g. of potassium chloride per 100 ml. of water) in a 500-ml. glass-stoppered bottle. Then 10 ml. of 0.3 *M* sodium periodate solution and 10 ml. of water were added and the mixture shaken at 25° for about one hundred hours. At this point the reaction was complete and ethylene glycol was added to consume the excess periodate. The formic acid present was titrated with 0.01 *N* barium hydroxide solution. One mole of formic acid was produced for 2.7 anhydrohexoside units. Samples of 200 mg. and 100 mg. gave similar values.

The presence of formic acid was confirmed by oxidation with mercuric chloride by the method of Auerbach and Zeglin.⁶ Formic acid (ca. 40 mg.) formed from a 400-

mg. sample of guaran was removed from the final reaction mixture by extraction with ether in a liquid-liquid extractor for ten days. A slight excess of sodium hydroxide was added to the ether extract and the mixture concentrated to about 5 ml. to remove ether and then was diluted to 60 ml. with water. After neutralization with 1 *N* hydrochloric acid, 1 ml. excess of acid and 3 g. of sodium acetate were added. The solution was filtered into an Erlenmeyer flask and 20 ml. of 5% mercuric chloride solution was added. The flask was covered with an inverted beaker and the mixture heated on a steam-bath for two hours. Precipitated mercurous chloride was filtered on a medium porosity sintered glass crucible, washed with hot water and ethanol, dried at 100°, and weighed. The weight of the precipitate corresponded to 103% of the formic acid determined by the above method of direct titration.

(6) F. Auerbach and H. Zeglin, *Z. physik. Chem.*, **103**, 161 (1922).

DEPARTMENT OF AGRICULTURAL CHEMISTRY

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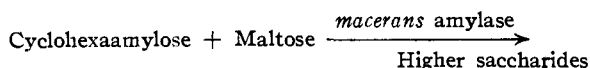
RECEIVED FEBRUARY 28, 1948

COMMUNICATIONS TO THE EDITOR

REVERSIBLE ACTION OF *macerans* AMYLASE¹

Sir:

The action of *Bacillus macerans* amylase² on starch has been interpreted by Cori³ as the exchange of a glycosidic bond in starch for a corresponding bond in a cyclic Schardinger dextrin (cycloamylose) molecule. In view of the small ΔF which would be expected for such an exchange, it might be expected that the reaction should be readily reversible. The reverse type reaction



has been tested with crystalline substrates and verified; *macerans* amylase thus has a synthetic as well as degradative action.

Pure cyclohexaamylose,⁴ 2.0 g., and c. p. maltose, 0.7 g., were dissolved in water, heated to complete mutarotation of the maltose, and treated with four units² of *macerans* amylase. The solution was made up to 100 ml. and the increase in rotation⁵ was followed in the polarimeter: initial rotation, 7.87°; after two hours, 8.10°. At this point the enzyme was inactivated by boiling and the reaction products separated by fractional precipitation. The least soluble fraction, 0.14 g., sirupy, had $[\alpha]_D +163^\circ$; average chain length by

alkaline ferricyanide,⁶ 8.9 glucose units. These values indicate that the sample is probably a mixture of saccharides containing some non-carbohydrate impurities. It gave a slight deepening of the color of I₂-KI solution and no unchanged cyclohexaamylose could be detected by the Tilden micro test.² On treatment with *macerans* amylase the fraction was rapidly reconverted in part into cyclohexaamylose as indicated by the formation of the characteristic I₂-KI complex.

Results indicating a similar synthetic action of *macerans* amylase have been obtained from cyclohexaamylose with glucose, α -methylglucoside, sucrose, cellobiose or maltobionic acid as co-substrates; also from cycloheptaamylose⁴ with maltose or glucose as co-substrates. These studies are being continued and will be reported in full at a later date.

(6) Levine, Foster and Hixon, *ibid.*, **64**, 2331 (1942).

CHEMISTRY SECTION
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RECEIVED AUGUST 10, 1948

FORMATION OF FLUORESCING SUBSTANCES FROM AMINO ACIDS

Sir:

Tauber¹ has reported recently on the formation of a fluorescing compound formed by the reaction of tryptophan with perchloric acid at room tem-

(1) Tauber, *THIS JOURNAL*, **70**, 2615 (1948).

(1) Journal Paper No. J-1581 of the Iowa Agricultural Experiment Station, Ames, Iowa. Project No. 964.

(2) Tilden and Hudson, *J. Bact.*, **43**, 527 (1942).

(3) Cori, *Federation Proc.*, **4**, 226 (1945).

(4) French and Rundle, *THIS JOURNAL*, **64**, 1651 (1942).

(5) McClenahan, Tilden and Hudson, *ibid.*, **64**, 2139 (1942).

perature. In the course of some preparative work we have noted the formation, also at room temperature, of striking blue fluorescing substances after treatment of tyrosine, phenylalanine and tryptophan with Denigès reagent.²

A fairly stable reagent was prepared by dissolving one part of paraformaldehyde in five parts of concentrated sulfuric acid. The reaction was carried out by dissolving a small sample in 2 ml. of sulfuric acid, adding 1 ml. of reagent and diluting with water after three minutes at room temperature. Fluorescence could be measured with the same filter combinations as are used for thiochrome determinations.

Under these conditions equal weights of the amino acids phenylalanine, tyrosine and tryptophan give intensities in the ratio of 100:4:1. If the acid solution containing tyrosine stood ten minutes before addition of reagent, no fluorescence was observed; this treatment did not affect the reaction with phenylalanine or tryptophan.

The fluorescing solutions are quite stable in the dark, less so in diffuse light, and lose about half their intensity after twenty minutes of ultraviolet irradiation.

Fluorescence is quenched by addition of bromine water, picric acid, hydrogen peroxide, or sodium sulfite, hydrosulfite or nitrite. Cysteine, but not cystine or methionine, inhibits the formation of the fluorescing compounds.

The reaction is also given by N-acetyltyrosine and ephedrine; not by alanine, arginine, asparagine, aspartic acid, cysteine, cystine, glutamic acid, glycine, histidine, isoleucine, lysine, methionine, proline, serine, threonine, valine, thiamine, riboflavin, niacinamide, pteroylglutamic acid, calcium pantothenate, rutin, dextrose, sucrose and maltose.

Crude substances have been isolated from reaction mixtures following treatment of phenylalanine or tyrosine, respectively. These substances react with ninhydrin (Van Slyke method) to yield carbon dioxide virtually equivalent to their total nitrogen content. This is contrary to what might be expected from the tetrahydroisoquinoline or carboline compounds formed with formalin by these amino acids under other conditions.³ Possibly a reversible condensation product of the triformal glycine ester type⁴ is formed. This would fit fairly well with the presently available analytical data on the tyrosine and phenylalanine compounds.

The relation between intensity of fluorescence and concentration of phenylalanine is practically linear over the range from 2–8 γ per ml. of diluted reaction mixture.

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RECEIVED AUGUST 25, 1948

(2) Denigès, *Compt. rend.*, **136**, 583 (1900).

(3) French and Edsall, "Advances in Protein Chemistry," Vol. II, Academic Press, New York, N. Y., 1945.

(4) Bergmann, *et al.*, *Z. physiol. Chem.*, **131**, 18 (1923).

REACTIONS OF IONS IN AQUEOUS SOLUTION WITH GLASS AND METAL SURFACES

Sir:

Radioactive tracers afford a more direct, sensitive and rapid means than any previously¹ used for studying the sorption of ions in solution on solid surfaces. Using them we have carried out exploratory investigations on some of the variables controlling such reactions. The method consists of immersing small flat samples in a solution of the radioactively tagged ion,² removing, rinsing and drying, and determining the intensity of the radioactivity on each with the aid of a Geiger-Mueller counter.

Soft glass samples immersed in 0.05 *M* sodium carbonate solution show a rapid sorption of sodium ions which approaches an apparent equilibrium value of about 0.5 "monolayers"³ at 25° and 5 monolayers at 90° after two to three hours immersion. The initial rate of sorption shows an approximately five-fold linear decrease with decrease in *pH* from 12 to 5.

Samples flamed prior to immersion show a much greater rate of sorption than those cleaned only by washing with water and vapor degreasing with carbon tetrachloride. The apparent activation energy⁴ was the same, however, *i. e.*, about 10,000 cal. mole⁻¹ in neutral solution. This suggests that the flame does not produce new types of reactive centers but uncovers more of the kind already available by cleaning the surface more completely than water and carbon tetrachloride alone. Presoaking of samples in 9 *N* HCl increased the activation energy for sorption in neutral solution to 13,500 cal. mole⁻¹ suggesting that the exchange of sodium ion with an —Si—OH bond may be more difficult than with an —Si—ONa bond.

Sodium ions sorbed on soft glass are removed only very slowly by rinsing in water at room temperature up to ten hours, but are removed somewhat more readily at higher temperature.

The sorption of silver and cesium ions is qualitatively similar to that of sodium ions. Presoaking of glass in stannous chloride solution, such as used in preparing glass for silvering, caused an increase in the sorption of silver ions.

From 0.2 to 5 monolayers of sodium ion are sorbed by cleaned aluminum, steel, silver and platinum during a few hours immersion at room temperature.

The sorption of carbonate ion on glass or steel is slight, of the order of 0.001 monolayer.

Radioautographs have been useful in determining the homogeneity of distribution of sorbed

(1) W. A. Weyl, "Some Practical Aspects of the Surface Chemistry of Glass," Glass Technology Institute, State College, Pa.

(2) U. S. Atomic Energy Commission Radioisotope Catalog No. 2.

(3) A "monolayer" is arbitrarily defined here as the number of ions required to cover the macro surface area of the sample if each ion covers an area equal to the square of its ionic crystal diameter.

(4) Calculated from the graph of the logarithm of the initial rate of sorption vs. $1/T$.

ions on surfaces and may be useful in practical testing of surface cleanliness.

This work has been supported by Wyandotte Chemicals Corporation and American Chemical Society fellowships and by Wisconsin Alumni Research Foundation funds allotted by the University Research Committee.

DEPARTMENT OF CHEMISTRY
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JAMES W. HENSLEY
ARTHUR O. LONG
JOHN E. WILLARD

RECEIVED AUGUST 13, 1948

SIDE-CHAIN CRYSTALLIZATION IN ALKYL POLYACRYLATES

Sir:

Rehberg and Fisher¹ synthesized and polymerized a series of *n*-alkyl acrylates from methyl up to hexadecyl. They reported that the brittle points of these polymers decrease with increasing length of side chain, up to octyl (-65°) after which further increase leads to higher brittle points (*e. g.*, $+35^\circ$ for hexadecyl polyacrylate). In the latter paper it is noted parenthetically that the higher alkyl polyacrylates are crystalline in character.

We have prepared polymers of tetradecyl, hexadecyl and octadecyl acrylate, and copolymers of hexadecyl acrylate with methyl acrylate, and have established the fact that the high brittle points of these polymers are due to crystallization of the *alkyl side chains*. At low temperatures these side chain crystallites bind the polymer into a firm, waxy structure. At a fairly sharp melting point the crystallites melt, and the polymer becomes rubbery. This phenomenon is distinct both from the second order transition phenomenon exhibited by the lower acrylates and from the usual type of polymer crystallization (alignment of the polymer chain itself).

A series of X-ray diffraction photographs were obtained from hexadecyl acrylate polymer which serve to confirm the side-chain crystallinity of these samples. Normal wide angle and small angle Debye-Scherrer patterns were taken. The wide angle diagrams showed the polymeric material to be crystalline, and exhibited a very strong line at 4.2 Å. Small angle patterns were obtained at film-to-specimen distances of 20 cm. with filtered Fe radiation. These diagrams exhibited two sharp diffraction lines at 50 and 150 Å., respectively.

A small angle diagram was also obtained from hexadecyl acrylate monomer. (This material is liquid at room temperature but the specimen was cooled so that it was crystalline.) This diagram showed a diffraction maximum at 28 Å. The length of the polymer side chain is somewhat less than this.

The 150 and 50 Å. spacings found in the polymer

(1) C. E. Rehberg and C. H. Fisher, *THIS JOURNAL*, **66**, 1208 (1944); *Ind. Eng. Chem.*, **40**, 1429 (1948).

may be considered as the first and third orders of a fundamental 150 Å. spacing. If the suggested interpretation of these long spacings as due to a crystallization of side chains is correct, then it would appear that the periodicity in the crystalline side chain packing repeats every six side chain units.

An effort was made to obtain orientation by drawing fibers of the hexadecyl acrylate polymer. X-Ray diagrams of these fibers showed slight orientation effects. The maxima of the long spacings occurred on the equator of these diagrams, which agrees with the interpretation that the crystallization is that of the side chains.

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RECEIVED JULY 20, 1948

(2) American Chemical Society Post-Doctoral Fellow 1947-1948.

POLAROGRAPHIC CURRENT TIME CURVES

Sir:

From theoretical considerations, Ilkovič¹ has shown that the average diffusion current for a dropping mercury electrode is given by

$$i_a = knD^{1/2}Cm^3/4t^{1/2} \\ = ICm^2/4t^{1/2}$$

If the "diffusion current constant" (I) for a given ion in a given supporting electrolyte is independent of capillary constant, as required by the Ilkovič equation, the performance of practical polarographic analyses can be greatly simplified. Lingane and Loveridge² have shown that, for the range of capillaries generally used in analysis, I is not strictly constant. The author recently has shown³ that it is not possible to properly correct a polarogram, obtained with a large series resistance, by simply subtracting the product of the observed average current and the series resistance from the applied e. m. f. In an attempt to compute the average current for a given applied e. m. f. and series resistance, he was unable to obtain agreement between the calculated and experimental values if he assumed the current during the life of a drop grew as the one-sixth power of the time.

It was, therefore, considered to be of importance both in practical analyses and theoretical studies to examine experimentally the current time relationship for individual drops.

The Brush Development Co. pen recorder, with which recordings can be made over a frequency range of d. c. to 100 c. p. s., was used to determine the relationship on the diffusion region for 0.001 *M* lead in 1 *M* potassium chloride (0.01% gelatin),

(1) D. Ilkovič, *Coll. Czech. Chem. Commun.*, **6**, 498 (1934).

(2) J. J. Lingane and B. A. Loveridge, *THIS JOURNAL*, **66**, 1425 (1944).

(3) H. A. McKenzie, unpublished work.

0.001 *M* cadmium in 0.1 *M* potassium chloride (0.01% gelatin), and oxygen in 0.1 *M* potassium chloride (0.01% gelatin). A hand fabricated capillary (1.63 mg.^{3/4} sec.^{-1/2}) and marine barometer tubing (2.05 mg.^{3/4} sec.^{-1/2}) were used. The current time curves were approximately one-third order parabolas with a slight discontinuity after about the first 0.3 sec. (drop time 0.4 sec.). The log *i* vs. log *t* plot was approximately a straight line in each case. The average slope was 0.29 and not 0.17 as required by Ilkovič. The ratio of maximum current to average current (determined graphically) had a mean value of 1.29 instead of 1.17. It is of interest to note that Schulman, Battey and Gelatis⁴ using a much slower recorder obtain a value of 1.25 for this ratio. Assuming the new current time relationship, the author was now able to accurately calculate the average current for a given applied e. m. f. and large series resistance.

In this preliminary communication, it is not possible to discuss the theoretical aspects of this study.

The author is deeply indebted to B. V. Hamon and the Electrotechnology Division of this Council for assistance. Grateful acknowledgement is due J. J. Lingane, M. C. Taylor and J. K. Taylor.

The above work forms part of the programme of Food Preservation Investigations of the Council.

(4) J. H. Schulman, H. B. Battey and D. B. Gelatis, *Rev. Sci. Instruments*, **18**, 226 (1947).

COUNCIL FOR SCIENTIFIC AND
INDUSTRIAL RESEARCH
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HUGH A. MCKENZIE

RECEIVED AUGUST 21, 1948

PHOTOBROMINATION OF METHYLSILOXANE FILMS ON GLASS SURFACES

Sir:

Methylchlorosilanes¹ are used for the production of hydrophobic films on glass and ceramic surfaces. It is supposed that molecules of the type Si(CH₃)₂Cl₂ are hydrolyzed to form a coating consisting of one or several layers of —Si(CH₃)₂—O—Si(CH₃)₂— chains with an exposed surface of —CH₃ groups.²

We have speculated that it should be possible to bring about photobromination, photochlorination or other reactions of the —CH₃ groups on the surface. Following halogenation still different functional groups might be introduced by processes such as the Friedel-Crafts reaction, thus altering the surface properties to meet specific requirements.

Our initial tests have demonstrated that the silicone surface can be photobrominated.

(1) Obtained for this work from the General Electric Co. under the designation Dri-Film 9987.

(2) Francis J. Norton, *Gen. Elec. Rev.*, **47**, no. 8, 6 (1944); Eugene G. Rochow, "Introduction to the Chemistry of the Silicones," John Wiley and Sons, Inc., New York, N. Y., 1946.

Test samples consisted of Pyrex and soft glass surfaces rubbed with the liquid silicone on a cotton swab. These specimens were exposed to radiobromine³ vapor at a pressure of about 10 mm. The air pressure in the flask was less than a millimeter; some water vapor was present. The samples were illuminated for one hour by a 1000-watt Mazda bulb placed about two inches from the flask. The surfaces treated with silicone picked up ten to thirty times the amount of bromine required for the monobromination of each methyl group in an estimated monolayer.³ Very little pickup occurred in the absence of illumination or with glass which was not coated. Rubbing with a dry towel after exposure reduced the bromine on the surface to a difficultly removable residue equivalent to approximately one monobrominated monolayer, but prolonged washing with water or carbon tetrachloride had relatively little effect. Illumination in the presence of chlorine gas for one hour removed a large fraction of the bromine. Dilute sodium hydroxide solution removed 50% of the bromine from some samples in fifteen minutes but showed much slower attack when the equivalent of only one or two monobrominated monolayers was left on the surface.

These results suggest the potentialities of radiohalogens as tools for investigating silicone films and furnish preliminary evidence that it may be possible to alter siloxane surfaces to meet specific requirements by substitution reactions, after the application of the silicone to the surface.⁴

This work was supported in part by the Research Committee of the Graduate School from funds supplied by the Wisconsin Alumni Research Foundation.

(3) Item No. 11 United States Atomic Energy Commission Radioisotopes Catalog No. 2. The specific activity when received was sufficient to give 20,000 counts per minute from one square inch of sample covered by an estimated monomolecular film of 5×10^{-9} moles of silicone² if each of the —CH₃ groups was converted to a —CH₂Br group.

(4) A more complete discussion of these experiments is contained in the senior thesis of G. L. Vandervort filed with the library of the University of Wisconsin in June, 1948.

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G. L. VANDERVORT
JOHN E. WILLARD

RECEIVED AUGUST 7, 1948

THE CONFIGURATION OF STREPTOSE

Sir:

Crystalline ethyl N-acetyldihydrothiostreptobiosaminide¹ was demercaptalated with aqueous mercuric chloride and the product hydrogenated with Raney nickel catalyst at 95° and 100 atm. for seven hours to yield the crystalline N-acetyl-tetrahydrostreptobiosamine, m. p. 78–80°, $[\alpha]^{26}_D - 104^{\circ}$ (*c* 0.838, water).

(1) F. A. Kuehl, Jr., E. H. Flynn, N. G. Brink and K. Folkers, *This Journal*, **68**, 2096 (1946).

Anal. Calcd. for $C_{15}H_{29}NO_{10} \cdot H_2O$: C, 44.88; H, 7.78; N, 3.49. Found: C, 44.70; H, 7.71; N, 3.33.

N-Acetyltetrahydrostreptobiosamine, on oxidation with two moles of periodate and subsequent acid hydrolysis gave N-methyl-L-glucosamine, one mole of formaldehyde, one mole of acetaldehyde and L-glyceric acid. The acid was separated from the mixture by low-pressure distillation as the methyl ester and was identified as the crystalline calcium salt dihydrate, m. p. 134–135° (dec.), $[\alpha]_D^{30} -12^\circ$ (*c* 2.60, water).

Anal. Calcd. for $(C_3H_5O_4)_2Ca \cdot 2H_2O$: Ca, 14.00. Found: Ca, 13.85. H. O. L. Fischer, *et al.*,² report $[\alpha]_D +12.9^\circ$ (*c* 5.19, water) for calcium D-glycerate dihydrate.

Since it has been shown that C2 and C3 of streptose have the same configuration,³ and that C4 has the L-configuration,⁴ then streptose is 3-C-formyl-L-lyxomethylose, a result in confirmation of the findings of Kuehl, Bishop, Flynn and Folkers⁵ whose work was based upon application

(2) E. Baer, J. M. Grosheintz and H. O. L. Fischer, *THIS JOURNAL*, **61**, 2607 (1939).

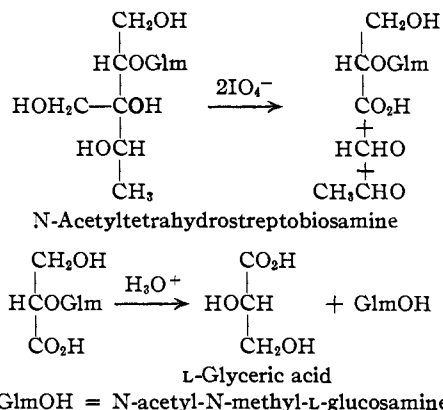
(3) N. G. Brink, F. A. Kuehl, Jr., E. H. Flynn and K. Folkers, *ibid.*, **68**, 2405 (1946); **70**, 2085 (1948).

(4) J. Fried, Doris E. Walz and O. Wintersteiner, *ibid.*, **68**, 2746 (1946).

(5) F. A. Kuehl, Jr., Mary Neale Bishop, E. H. Flynn and K. Folkers, *ibid.*, **70**, 2613 (1948).

of the hydrazide rule to polarimetric data. This evidence also represents an independent proof for the skeletal structure of streptose and the point of attachment of N-methyl-L-glucosamine thereon, since there is only one structure that would give the products obtained.

The reactions involved may be represented as



GlmOH = N-acetyl-N-methyl-L-glucosamine

DEPARTMENT OF CHEMISTRY
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RECEIVED JULY 29, 1948

(6) Bristol Laboratories Research Fellow of The Ohio State University Research Foundation (Project 224).

NEW BOOKS

Fatty Acids, Their Chemistry and Physical Properties.

By KLARE S. MARKLEY, Principal Chemist, Oil, Fat and Protein Division, Southern Regional Research Laboratory, U. S. Department of Agriculture, New Orleans, La. Interscience Publishers, Inc., 215 Fourth Ave., New York 3, N. Y., 1947. x + 668 pp. 81 figs. 15.5 × 23.5 cm. Price, \$10.00.

Throughout the vigorous development in most branches of organic chemistry, the field of fatty compounds did not attract much interest until the last two decades when the fatty acids and their derivatives have been subjected to an intensive study. Physico-chemical and physical techniques are now employed in this field perhaps more extensively than in many other branches of organic chemistry.

As a result of the ever-increasing development of appropriate tools and methods, a vast quantity of information has been produced by workers approaching the various problems from different angles. The literature, both old and new, is widely scattered and consequently there is an urgent need for a comprehensive, well-organized book which adequately presents these modern developments, and often unrelated data, in integrated form.

Klare S. Markley's book is the first and successful attempt to adequately cover the field. The material is grouped into six main chapters, dealing with the nature and history of fats and waxes, classification and structure of the fatty

acids, their physical properties, chemical reactions, synthesis, and the isolation and identification of fatty acids. The following topics, picked at random, may indicate the scope of the book: isomerism, X-ray diffraction, polymorphism, spectral properties, esterification, hydrogenation, oxidation, biological oxidation, nitrogen derivatives, *in vitro* synthesis, biosynthesis. The author, in his endeavor to give a comprehensive picture of the subject matter, broadened the scope so that, in addition to the presentation of the fatty acids, he also discussed derivatives whenever it seemed desirable. It is evident, from his presentation, that physico-chemical and physical means are to an appreciable extent responsible for the new development in the field of fatty acids.

The interest in fatty acids is rapidly broadening and the number of research men working in this field is increasing accordingly. Much of the existing information is being corrected or modified and a large quantity of new information is constantly being produced so that a revised edition of the present book may become necessary in the not too distant future. The subject matter may then have grown to such dimensions that the author may find it expedient to have individual chapters in his book written by specialists in the respective fields.

Markley's monograph will prove useful in the hands of every chemist, physicist, biochemist, medical man and technologist engaged in studies dealing with fatty acids and their derivatives. The author has succeeded in pre-